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Items of interest from our laboratory notebooks

▶ Most analysts know about 1:10-phenanthroline and many use it for iron determinations. Not so many people seem to know that **4:7-diphenyl-1:10-phenanthroline** is twice as sensitive as 1:10-phenanthroline in the colorimetric determination of iron. There are several papers on the subject but the latest is *Analyst*, 1958, **83**, 80. The reagent is also called **Bathophenanthroline**, and we make it.

▶ Then, again the substitution of methyl groups in the 2:9 positions has the interesting effect of making the reagent insensitive to iron and we then have a selective and sensitive reagent for copper (see *Anal. Chem.*, 1956, **28**, 1158). Hopkin & Williams make **2:9-dimethyl-1:10-phenanthroline** (sometimes called **Neocuproin**).

▶ One does not think of sulphate as a radical one can determine absorptiometrically, but this is now possible for low concentrations. **Barium chloranilate** is the reagent and there are two papers on the subject—*Anal. Chem.*, 1957, **29**, 281 and *Anal. Chem.*, 1958, **30**, 202. Hopkin & Williams make it.

▶ Hopkin & Williams Ltd. were also early off the mark with supplies of the remarkable new colour-producing reagent for fluoride ions, **3-aminomethylalizarin-NN-diacetic acid**, described by Belcher, Leonard and West (*Talanta*, 1959, **2**, 92.) This important reagent is already available from stock.

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

853. **Sodium tetraphenylboron, 1958: a biblio-phy.** A. J. Barnard, jun., and H. Büechl (J. T. er Chem. Co., Phillipsburg, N.J., U.S.A.). *mist Analyst*, 1959, **48** (2), 44, 45 and 49.—This tains 54 references for 1958 and 27 additional rences for 1955, 1956 and 1957.

854. **Applications of sodium tetraphenylboron.** . Teygeler (Rijksinst. voor Pharmacotherapeut. lerzoek, Leiden, Netherlands). *Chem. Pharm. h.*, 1957–8, **13**, 48, 57, 75, 90.—The applications Na tetraphenylboron in the identification of a ıber of organic bases, the purification of sodium s and the quant. determination of K are ewed. (45 references.) M. J. MAURICE

855. **Introduction to uses of EDTA.** H. J. ey (Gen. Electric Co., Wembley, England). *oc. Glass Tech.*, 1959, **43**, 30T–35T.—A general ription is given of the use of EDTA for (*i*) direct titration of Ca, Mg, Ba, Al, Zn, Pb, etc., the sequestration of interfering metals in the rmination of Mo, W, U, Be, Ca, Ba, K, Cu, $^{3-}$ and B_2O_3 and (*iii*) the control of oxidation - ction equilibria in polarography.
J. A. SUGDEN

856. **EDTA as a micro-analytical reagent.** . Barnard, jun., W. C. Broad and H. Flaschka . Baker Chem. Co., Phillipsburg, N.J., U.S.A.). *ochem. J.*, 1959, **3** (1), 43–64.—A literature ey is given, with 196 references, covering (*i*) the of EDTA as a masking agent in quant. pptn., ometric determinations, solvent extractions, tions, and qual. analysis, (*ii*) EDTA micro- tions, (*iii*) the use of EDTA as a chromogenic solubilising agent, (*iv*) its use in chromato- hic and ion-exchange procedures, and (*v*) ellaneous applications to polarography, electro- sition and electro-analysis, the polarimetric mination of sugars, the stabilisation of ascorbic soln., and the removal of metals from com- ial ammonia gas. R. E. ESSERY

857. **Indicators for non-aqueous acid - base titri-y. II.** J. T. Stock and W. C. Purdy (Univ. aecticut, Storrs, Conn.). *Chemist Analyst*, 1958,), 50–56.—A review with 124 references.

858. **New acid - base indicator: (1:3:4-iiazol-2-yl)-2-naphthol.** D. I. Kurbatov and N. azarinova. *Izv. Sibirsk Otd. Akad. Nauk SSSR*, . (8), 94–97; *Ref. Zhur., Khim.*, 1959, (7), r. No. 22,948.—With increasing pH of the ator soln. the colour changes from pale yellow nk at pH 7·39. K. R. C.

3859. **Complexometric indicators.** A. I. Busev and A. G. Petrenko. *Zavod. Lab.*, 1958, **24** (12), 1449–1457.—A review with 163 references is presented. G. S. SMITH

3860. **Metomega chrome cyanine [Solochromate fast blue B, C.I. Mordant Blue 7] as a metal indicator in EDTA titrations.** A. A. Abd El Raheem and M. M. Dokhana (Min. of Public Health Lab., Cairo, Egypt). *Anal. Chim. Acta*, 1959, **20** (2), 133–136.—The blue colour of a solution of C.I. Mordant Blue 7 changes to pink or red when it is added to solutions of various metal ions. The identification limits and dilution limits of 16 metal ions are given but the reaction is not specific. The dye, used as a mixture with 100 parts of NaCl, serves as an indicator for the EDTA titration of Mn, Cd, Zn and Pb (*cf.* Belcher *et al.*, *Chemist Analyst*, 1957, **46**, 86). The soln. is buffered at pH 6·8 with a mixture of 0·1 *M* barbitone sodium and 0·1 *N* HCl (522 : 478) and is titrated with 0·01 *M* EDTA (disodium salt). H. N. S.

3861. **Chelatometry. XVII. The use of 4-(2-pyridylazo)resorcinol (PAR), thiourea and tri(hydroxymethyl)methylamine.** P. Wehber (Chem. Lab. d. Elektrizitätswerk Wesertal G.m.b.H., Hameln, Germany). *Z. anal. Chem.*, 1959, **166** (3), 186–189.—Calcium, Cu^{2+}, Zn^{2+}, Cd^{2+}, Hg^{2+}, Pr^{3+}, Nd^{3+}, Pb^{2+}, Bi^{3+}, Mn^{2+}, Ni^{2+} and Al^{3+} may all be determined by titration with EDTA, with 4-(2-pyridylazo)resorcinol (**I**) as indicator. In a tri(hydroxymethyl)methylamine buffer (pH 9 to 9·5), Cd^{2+}, Hg^{2+}, Mn^{2+} and Pb^{2+} may be determined with **I** as indicator, Mn^{2+} with Eriochrome red B (C.I. Mordant Red 7) as indicator, and Co^{2+} with murexide as indicator. Masking and titration procedures are reported for thiourea.
T. R. ANDREW

3862. **Tervalent molybdenum as volumetric reducing agent.** M. Y. Farah and S. Z. Mikhail (Atomic Energy Estab., Cairo, Egypt). *Z. anal. Chem.*, 1959, **166** (1), 24–31 (in English).—Soln. of $MoCl_3$ can be prepared by the electrolytic reduction of molybdic acid in 8 *N* HCl or by reduction with metallic mercury in 9 *N* HCl. In acid soln. $MoCl_3$ has been used for the titration of Ce^{4+}, $Cr_2O_7^{2-}$, Fe^{3+}, VO_3^-, IO_3^- and BrO_3^-; the max. error is 1% with potentiometric end-point determination. The soln. are sufficiently stable for use in an open burette, but air must be excluded from the titration vessel. G. BURGER

3863. **Spectrofluorimetry and its application to chemical analysis.** R. J. Bartholomew. *Rev. Pure Appl. Chem., Australia*, 1958, **8** (4), 265–301.—A review.

3864. **Radiometric titration using solutions of cobalt-60 complexes.** K. B. Yatsimirskiĭ and E. N.

Roslyakova. *Trudy Komiss. Anal. Khim.*, 1958, **9** (12), 194–199; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 27,008.—Large anions (PO_4^{3-}, SO_4^{2-} and MoO_4^{2-}) were determined by their radiometric titration with hexa-amminocobaltic chloride soln. containing ^{60}Co. The end-point was determined from two measurements corresponding to two points on the titration curve (Langer, *Anal. Chem.*, 1950, **22**, 1288). In the determination of PO_4^{3-}, the test soln. was titrated with hexa-amminocobaltic chloride soln. at pH $\simeq$ 13; CrO_4^{2-}, $Fe(CN_6)^{3-}$, $Fe(CN_6)^{4-}$, $C_2O_4^{2-}$, SO_4^{2-}, CO_3^{2-}, F^-, Br^-, I^-, tartrates and citrates interfered. The titration of SO_4^{2-} was carried out in neutral or slightly acid soln. in the presence of ethanol ($<$ 50% of the total vol. of test soln.); large amounts of NO_3^- interfered. Molybdate was pptd. in an acetic acid medium at pH 3 to 4; WO_4^{2-} and VO_3^- interfered. The relative error of the determination did not exceed $\pm$ 2% with a content of 3 to 10 mg of PO_4^{3-}, 1 to 10 mg of SO_4^{2-}, and 0·6 to 31 mg of MnO_4^{2-}. The method for the determination of PO_4^{3-} was also used for the determination of P_2O_5 in phosphorite. K. R. C.

3865. **Radioactive isotopes in the separation of elements by means of anionites.** A. A. Pozdnyakov and A. K. Lavrukhina. *Trudy Komiss. Anal. Khim.*, 1958, **9** (12), 161–169; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 26,991.—A method for the separation of Ni^{2+}, Mn^{2+}, Cu^{2+}, Fe^{3+} and Zn^{2+}, based on the formation by these elements of chloride complexes in HCl or LiCl soln., was developed; radio-isotopes were used to follow the course of the separation. The test soln. (0·3 to 0·5 ml), made 12 *M* with HCl and containing each of the elements mentioned (5 to 10 mg), was introduced into a column. (10 cm $\times$ 0·4 cm), filled with the anionite Dowex I-X8, which had previously been brought into equilibrium with 12 *M* HCl. The most effective separation was observed with a succession of elutions with HCl at different concn.: 12 *M* HCl for Ni, 6 *M* for Mn, 4 *M* for Co, 2 *M* for Cu, 0·5 *M* for Fe and 0·01 *M* for Zn. The separation took $\simeq$ 3 hr. A much more rapid but somewhat less efficient separation of the elements was obtained if a soln. in 12 *M* LiCl was passed through a column of anionite saturated with 12 *M* LiCl followed by elution with H_2O at a temp. of 80°. EDTA (**I**) was used as the complexing reagent in the separation of Ca from Sr; the test soln. of the **I** complexes of Ca and Sr was passed through a column of anionite in the EDTA form, and the components were then eluted with 0·1 *M* **I** at pH 11·2. The separation of Fe^{3+}, Cr^{3+} and VO^{2+} should precede the heating of the test soln. with **I** for the purpose of reducing V^{5+} to VO^{2+} and for the rapid formation of the **I** complex of Cr. The cooled soln. (0·1 to 0·2 ml) was passed through the column of EDTA resin, which was then washed through with a 0·1 *M* soln. of **I**; Fe was eluted more rapidly than Cr. A very efficient separation of a mixture of Ce, Nd, Er and Yb is obtained by the introduction of a soln. of the **I** complexes of these elements (0·1 ml) into a column of EDTA resin followed by elution of the individual components with 0·13 *M* citric acid. Elution with a soln. of **I** is no guarantee of satisfactory separation. An effective method of separating ^{90}Sr and ^{90}Y is the adsorption of the **I** complexes of both elements by the EDTA anionite SN-1 followed by elution of ^{90}Sr and then of ^{90}Y by a 0·01 *M* soln. of **I** at pH 8. K. R. C.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

3866. **Analysis for industry [determination silicon, arsenic and germanium].** A. M. Macdonald. *Ind. Chem.*, 1959, **35**, 142–144 The literature on the application of organic-b molybdates to this determination, *e.g.*, of Ge pyridine molybdogermanate, is reviewed. references.) O. M. Whitton

3867. **Spectrographic analysis of micro amou of gas.** O. P. Bochkova, L. P. Razumovska and S. E. Frish. *Optika i Spektroskopiya*, 1958 (5), 624–626; *Ref. Zhur., Khim.*, 1959, (8), Abs No. 27,105.—The method was used for the det mination of O, N and A in very small amounts air. To obtain the necessary pressure in the d charge tube the test air (250 ml) was evacuated w a Töpler pump (1 $\times$ 10^{-4} mm of Hg in a capillary diam. 0·5 mm) or inert gas, such as He, was add Spectra were excited and calibration curves w constructed with co-ordinates of ΔS, and log for the lines 7772 (O) *vs.* 7468 (N) and 7503 (A) 7468 (N) lines. A change in concn. of A did affect the determination of O, but a change concn. of O caused a shift in the A spectru The mean error of determination for O was $\pm$ 15 and for A $\pm$ 8%. The addition of He (in 3 100-fold amounts) increased the sensitivity of t test, but an increase in the mass of gas decreas it, this effect being due to a change in compositi of the mixture arising from adsorption and d sorption of the gas by the walls of the discha tube. When He was added the amount of sam could be decreased to 0·01 cu. mm at atmosphe pressure; the error of determination for O w then $\pm$ 12% and for A $\pm$ 20%. K. R. C.

3868. **Isotope dilution as a method for the det mination of some rare elements.** I. P. Alima and G. N. Bilimovich. *Trudy Komiss. Anal. Khi* 1958, **9** (12), 219–225; *Ref. Zhur., Khim.*, 1959, Abstr. No. 26,988.—The conditions were studi for the separation of Nb - Ta, Nb - Zr and Ta - mixtures by their pptn. with ammonium benze seleninate (**I**) at various pH levels, followed their determination as benzeneseleninates by i tope dilution, with the radio-isotopes ^{95}Nb, ^{182} and ^{95}Zr. A 10% soln. of **I** was used for the ppt the ppt. formed were ignited to oxides at 800° 900°. The effect of the presence of some hydro acids was studied. It was shown that more efficie pptn. of Nb and Ta from a citric acid medium w obtained by the addition of mineral acids; t addition of mineral acids to oxalic acid soln. b no effect, and in the presence of trihydroxyglut acid no Nb or Ta was pptd. by **I**; in an ascor acid medium an orange ppt., which quant. remov Ta and could be used for the concentration small amounts of Nb and Ta, was produc instantaneously by the ascorbic acid reduction **I**. A detailed study was made of the effect of citric acid concn. with different concn. of HCl the degree of pptn. of Nb and Ta. It was sho that 60% of Ta and 10% of Nb were pptd. fr a soln. containing 2·5% of citric acid and 15% HCl. The results of determinations by isoto dilution methods and by β-ray reflection meth were in good agreement. The separation of Nb - Zr mixture by means of **I** was carried ou

presence of 0·5% of citric acid and 15% of ; the most accurate results were obtained for tio of Nb to Zr of 1:25. The separation of Ta n Zr (optimum ratio of Ta to Zr of 1:50) was ied out from a soln. containing 25% of citric and 15% of HCl; the final concn. of **I** was 1·5%. purity of the recovered materials was tested the constancy of the specific activity of the npound. Electron micrographs of the ppt. of and Ta with **I** are given. K. R. C.

869. **Spectrographic determination of trace urities in solutions (on metal electrodes in a rk).** J. Czakow, T. Steciak and O. Szczerbinska lish Acad. Sci., Inst. Nucl. Res., Warsaw). . NP–7085, issued by U.S. Atomic Energy nm., 1958, 12 pp. (English translation).—The lication of the "copper spark" method to the ermination of trace amounts of Al, As, Be, B, Ca, Ce, Cr, Cu, Fe, La, Pb, Mg, Mn, Ni, P, Si, Na, Te and Sn in soln. of isotopes of low ioactivity and containing PO_4^{3-}, SO_4^{2-} and I^- iven. The calculated coeff. of variation for ΔS a soln. with a concn. of 1 μg of Ni per ml was 5% for 95% accuracy. A purification method the auxiliary electrode and the use of the ndard paper density scale for quant. deter- nations are described. Nucl. Sci. Abstr.

870. **Analysis of ternary alloys [of antimony, minium and gallium].** Yu. A. Chernikhov and V. Cherkashina. *Zavod. Lab.*, 1959, **25** (1), 27.—For Sb, the alloy is dissolved in H_2SO_4 - $(NH_4)_2SO_4$ and the soln. is titrated with $KBrO_3$ n. For Al and Ga, the sample (0·05 to 0·1 g) dissolved in 15 to 20 ml of dil. HCl (1:1), with addition of HNO_3, and Sb is oxidised by a ll amount of solid $KBrO_3$. The soln. in a arating-funnel is shaken with an equal vol. of yl acetate for 1 min. to extract the Ga and st of the Sb. The extract is separated and hed with 3 to 4-ml portions of dil. HCl and lined. The aq. soln. is evaporated to dryness, residue is dissolved in 50 to 60 ml of water, the soln. is mixed with 1·5 times the theoretical unt of EDTA (disodium salt), then neutralised Congo red indicator with aq. NH_3 (1:4), mixed n 5 ml of 2 *M* chloroacetic acid, 9 ml of *N* Na ate and 1·5 ml of 0·1% alizarin red S soln., titrated for Al with 0·02 or 0·05 *M* $Th(NO_3)_4$ change from yellow to red. For the determina- of Ga, the retained extract is twice shaken rously for several minutes with 15 to 20 ml % tartaric acid soln. The combined aq. layers neutralised to methyl red indicator with aq. , and solid hydroxyammonium chloride is d to destroy $KBrO_3$. A known amount (2 to in excess) of 0·01 *M* EDTA (disodium salt) some solid Eriochrome black T - NaCl mixture 00) are added, followed by aq. NH_3 (1:2) to a bright blue colour, and neutralisation with HCl (1:1) to give a pink colour. The pH is e $\simeq$ 8 by dropwise addition of an ammoniacal r soln. (pH 10) until the soln. turns blue and a further 1 or 2 drops and $\simeq$ 100 mg of Cl are added. The excess of EDTA is back- ted with a soln. of Zn until the colour changes iolet. The methods were found satisfactory lloys containing 65 to 81% of Sb, 0·8 to 16% and 2·9 to 34% of Ga. G. S. Smith

871. **Studies on extraction with *iso*butyl methyl ue and its use for the determination of metal I. Extraction of iron, antimony, tin, arsenic, selenium, tellurium and germanium from an acid solution.** Hidehiro Gotô, Yachiyo Kakita and Takeshi Furukawa (Inst. Iron, Steel and other Metals, Tohoku Univ., Katabira-cho, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (12), 1513–1520.—The extraction of metals with *iso*-butyl methyl ketone was examined in mineral acids of various concn. and the results are shown schematically. From an HCl soln. > 5·5 *N*, Fe^{III} and Sn^{II} are quant. extracted, and the presence of other acids (*e.g.*, H_2SO_4 and $HClO_4$) facilitates the extraction. Bivalent Fe is not extracted, but Sb^{V}, Sb^{III}, Sn^{IV} and Se^{IV} are quant. extracted from HCl soln. (> 8 *N*). In the presence of 10 *N* H_2SO_4, 0·5 *N* HCl suffices for complete extraction of Sb^{V} and Sb^{IV}. Extraction of As^{III} is $\simeq$ 90% in 8 *N* HCl with one extraction, whilst that of As^{V} is $\simeq$ 25% irrespective of the concn. of HCl. About 96% of Te^{IV} and $\simeq$ 98% of Ge^{IV} are extracted from $\simeq$ 8 *N* HCl. No interference results from H_2SO_4 and $HClO_4$ in the extraction of As^{III}, Se^{IV}, Te^{IV} and Ge^{IV}. In the absence of Cl^-, none of these is extracted.

II. Extraction of chromium, vanadium, molybdenum and manganese from an acid solution. Yachiyo Kakita and Hidehiro Gotô. *Ibid.*, 1958, **79** (12), 1520–1524.—Similar experiments were carried out with Cr^{VI}, V^{V}, Mo^{VI} and Mn^{VII}. Only a small amount of V (< 10%) is extracted from a mineral acid soln. Extraction of Cr^{VI} ($\simeq$ 82%) is best effected from a 6 *N* HCl soln.; in HCl > 8 *N*, Cr^{VI} is reduced to Cr^{III}. Extraction from H_2SO_4 (1 to 10 *N*), HNO_3 (2) to 5 *N* and $HClO_4$ (2 to 5 *N*) is less efficient. About 95% of Mo^{VI} is extracted from 8 *N* HCl. The addition of $HClO_4$ to the HCl soln. facilitates the extraction of Mo and Cr. Only a small proportion (< 32%) of Mn^{VII} is extracted (optimum acidity, 9 *N* H_2SO_4).

K. Saito

3872. **Quantitative radiochemical analysis by ion exchange. Anion-exchange behaviour of several metal ions in hydrochloric, nitric and sulphuric acid solutions.** L. R. Bunney, N. E. Ballou, J. Pascual and S. Foti (U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 324–326.—To facilitate possible separations of Am, Ce, Mo, Pa, Nb, Ru, Sr, Th, U, Y and Zr by anion exchange, the partition coeff. (K_d) between their soln. in HCl, HNO_3 or H_2SO_4 and the resin Dowex 2 were studied and K_d plotted against acid normality. Strontium, Y, Ce and Am were not appreciably adsorbed by any of these 3 systems; Th, Pa and U may be sequentially separated by conc. HCl, 4 to 5 *N* HCl and 0·1 *N* HCl; U is separated from Mo with 0·1 *N* HCl. By using 8 *N* HNO_3, U, Zr and Th are eluted in that order; Am (or rare-earth metals) is separated from Th, U, Pa and Zr in 0·1 *N* H_2SO_4; Th may then be eluted with 2 to 4 *N* H_2SO_4. Further separations are possible by use of the combined acids. The behaviour of the resins Dowex 1 and 2 was compared.

P. D. Parr-Richard

3873. **Quantitative radiochemical analysis by ion exchange. Anion-exchange behaviour in mixed acid solutions and development of a sequential separation scheme.** L. Wish (U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 326–330.—The rapid separation of isotopes of Np, Pu, U, Zr, Nb and Mo is possible by ion exchange on Dowex-2 resin from an HCl - HF medium; distribution coeff.

between the resin and HCl - HF soln. of the isotopes were measured. The effect of variations in HF concn. is discussed. Quadrivalent Zr, Nb^{V} and Mo^{VI} are adsorbed strongly from 0·1 *N* HCl; Np^{IV}, Pu^{IV} and U^{VI} much less strongly. Neptunium is separated from Nb with 6·5 *N* HCl - 0·004 *N* HF, and Nb eluted with 6·0 *N* HCl - 0·3 *N* HF; U is separated from Mo with 0·1 *N* HCl - 0·06 *N* HF, and Mo is then eluted with 12 *N* HNO_3. The activities so eluted are quant. determined in a γ-ray scintillation well counter or a 256-channel γ-ray spectrometer. Molybdenum, Np and Pu are obtained almost pure; Zr and Nb contain γ-ray impurities which do not interfere with the determination of these elements. Uranium is contaminated with ^{132}Te and ^{132}I, making its determination difficult. P. D. PARR-RICHARD

3874. **Quantitative radiochemical analysis by ion exchange. Anion-exchange equilibrations in phosphoric acid solutions.** E. C. Freiling, J. Pascual and A. A. Delucchi (U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 330–331.—Distribution coeff. between Dowex-2 resin and H_3PO_4 soln. of Cs, Sr, Ce^{III}, Ce^{IV}, Zr^{IV}, Te^{IV}, Np^{IV}, Nb^{V}, Mo^{VI} and U^{VI} were measured. Caesium and Te are not adsorbed even from 0·1 *N* acid, but Sr, Ce^{III} and Ce^{IV} are weakly adsorbed at low acid concn. and the other elements are all strongly adsorbed. Thus Te can be separated from U obtained by a sequential separation scheme previously described (*cf.* Wish, *Anal. Abstr.*, 1959, **6**, 3873). P. D. PARR-RICHARD

3875. **Determination of argon and oxygen by gas chromatography.** G. S. Vizard and A. Wynne (Nat. Coal Board, N.E. Div., Sci. Dept., Parkgate, Rotherham, England). *Chem. & Ind.*, 1959, (6), 196–197.—Up to 0·05 ml of A can be separated quant. from O by passage through a column of Linde Molecular Sieve No. 5A (30 to 60 mesh), activated at 400° for 1 hr. The hot material is packed in a glass column (10 metres × 4 mm int. diam.); H as carrier gas gives a more sensitive katharometer response than N. A by-pass pipette system is used for sample injection; peak broadening in relation to pipette size is discussed. The temp. of operation is 18°. P. D. PARR-RICHARD

3876. **Spectrographic determination of lithium in nuclear-grade calcium.** J. Artaud and J. Cittanova. *Comm. Énergie Atomique, France*, Rapp. No. 676, 1957, 7 pp.—The metallic samples are converted directly into the carbonate by addition of ammonium carbonate. A method of fractional distillation in the arc, by using KCl as carrier, allows determination of the Li content to within 0·1 p.p.m. CHEM. ABSTR.

3877. **Isotopic analysis of lithium by mass spectrometry.** P. J. Bentley, J. Bishop, D. F. Davidson and P. B. F. Evans (U.K. Atomic Energy Authority, Res. and Dev. Branch, Capenhurst, Chester). *J. Sci. Instrum.*, 1959, **36** (1), 32–34.—The instrument described is provided with two ion sources by having three arms in the spectrometer tube, one for the collector, the others for the sources. Each source is fitted with a vacuum lock to reduce pumping times. The ^{6}Li to ^{7}Li ratio is measured automatically by scanning the magnetic field so that each ion beam is brought alternately on to the collector, where it is amplified and "memorised" as a voltage on a capacitor. The amplifier sen tivity is adjusted for one beam until the t voltages are equal. The isotope ratio is given the ratio of the amplified sensitivities for the t beams. Analyses can be made at a continuo rate of one every 20 min. G. SKIRROW

3878. **Spectrographic determination of small co centrations of sodium in solutions [of aluminiu** N. M. Drichko. *Sb. Rabot Stud. Nauch. Obsh Leningrad. Inst. Tochnoĭ Mekhan. i Optiki*, 19 (35), 45–49; *Ref. Zhur., Khim.*, 1959, (7), Abs No. 23,015.—Aluminium shavings were dissolv in HCl and 3 drops of the soln. were transferred a carbon electrode which had received a prelimina purification in an a.c. arc discharge at 20 amp. 1 min. Spectra were excited at 1·5 amp. in low-voltage spark discharge with a capacity 6 μF. Calibration curves of the 5889·9-A line Na were constructed of log (I_{Na}/I_F) *vs.* log The minimum Na concn. determined with respe to Al was 0·0025%. The analysis was carried o with a Zeiss spectrograph Gl-12, and panchroma plates. The slope of the calibration curve was 0·9. With an arc discharge the slope of the ca bration curve was 0·5. K. R. C.

3879. **Determination of sodium, potassium a lithium in silicates by flame photometry.** I. Voinovitch and J. Debras (Soc. Française Céram., Paris). *Ind. Céram.*, 1958, (502), 32 327.—The sample is decomposed with conc. H_2S plus HF, the soln. is evaporated to dryness a the residue is dissolved in dil. HCl at ≃ 10 Sodium, K and Li are then determined simultan ously after removal of Al, Fe, Ti and the bu of the Ca by co-pptn. at 15° to 25° with aq. N and ammonium carbonate in the presence $(NH_4)_2SO_4$. Line intensities are measured 5910 A (Na), 7700 A (K) and 6720 A (Li), inte ference filters being used. The method is rapi and accuracy and reproducibility are high. W. J. BAKER

3880. **Rapid determination of alkali met [sodium and potassium] in baryta glass.** A. Zan'ko and N. D. Levina. *Nauch. Zap. L'vov Politekh. Inst.*, 1958, (50), 57–62; *Ref. Zhur., Khi* 1959, (6), Abstr. No. 19,085.—*Procedure*—Tr the finely powdered glass (0·5 g) in a platinum d with 40% HF (10 ml), heat in an air bath at 1 to 140° until the excess of HF is completely moved, add hot H_2O (20 to 25 ml) and pher phthalein (1 drop), cover the dish with a wat glass, add freshly prepared CaO (1·5 g) carefu and gradually, heat for 1 hr. with occasio mixing, add $Ba(OH)_2$ soln. (4 to 5 drops) precipitate SO_4^{2-} and heat for 5 to 10 min. Fi off the ppt. and wash 10 to 15 times with a soln. of CaO (1·5 g) in H_2O (500 ml). Satu the filtrate and washings with CO_2 until the s is decolorised, heat until a colour appears and t boil for 5 to 10 min. until the bicarbonates completely decomposed. Quickly filter off $CaCO_3$ residue, wash with cold H_2O (0·5 to 1 portions), rapidly cool to room temp. and tit the K_2CO_3 and Na_2CO_3 with 0·1 *N* HCl, with me orange as indicator; add an excess of HCl (2 ml) to this soln., evaporate to dryness on a w bath, dry the residue of NaCl plus KCl for 30 at 150°, dissolve it in H_2O (1 to 2 ml), filter off C and SiO_2, evaporate the filtrate and wash to 0·1 to 1 ml and while mixing add 0 magnesium - hexanitrodiphenylamine (**I**) (15

ter off the ppt., wash (× 2 or 3) with a cold d. soln. of potassium - **I** and then with H_2O, l to 0° and dry for 1 to 1·5 hr. at 100° to 110° l weigh. Sodium is calculated by difference. o methods are suggested for the control. In e, the control is carried through all the analytical ges except titration with HCl up to the production pure soln. of NaCl and KCl, in aliquots of which is determined by **I** and Na by the uranyl acetate thod. In the second method, Na_2CO_3 is dissolved H_2O (25 ml), HF (10 ml) is added and the test continued as for the main test, including the ermination of K. The whole determination es 6 to 8 hr. K. R. C.

3881. **Temperature conditions for the colorimetric determination of potassium in the form $Pb[Ni(NO_2)_6]$.** A. I. Krupkin and V. L. novalov. *Zavod. Lab.*, 1958, **24** (12), 1444.—e temp. of pptn. of the complex $K_2Pb[Ni(NO_2)_6]$ uld not exceed 30° and should be kept the same all soln. G. S. Smith

3882. **A new micro-gasometric method for the ermination of potassium.** N. Ganchev (Higher st. Nat. Economy, Varna). *Izv. Khim. Inst., lg. Akad. Nauk*, 1958, **6**, 149–166.—Potassium d accompanying metals (Na, Ba, Mg or Ca) are nverted into perchlorates and extracted with yl acetate (satd. with $KClO_4$). The undissolved ClO_4 is washed and heated and the evolved O asured. In the presence of 50 times the quantity Na or Ba the mean error is ± 1%. N. E.

3883. **Determination of potassium oxide in glass.** J. Cluley (Gen. Electric Co., Wembley, England). *Soc. Glass Tech.*, 1959, **43**, 62T–72T.—The thod of pptn. as K tetraphenylboron (*cf.* Cluley, *alyst*, 1955, **80**, 354) is made more rapid by ding a known excess of Na tetraphenylboron soln., l back-titrating the excess with $Hg(NO_3)_2$ soln. e determination is completed within 2 hr. and ults agree within 0·1% with those from gravitric determinations. J. A. Sugden

3884. **Solubility of caesium metaperiodate and co-precipitation in the determination of potassium the metaperiodate.** E. Snyder and R. J. Robinson em. Dept., Univ. of Washington, Seattle, .A.). *Anal. Chem.*, 1959, **31** (3), 474–475.—assium has been determined by pptn. as the aperiodate from 33% (v/v) ethanol buffered at 3·5, followed by iodimetry (*cf.* Jentoft and binson, *Anal. Abstr.*, 1957, **4**, 1760). The ium salt is too soluble for the determination of under the same conditions. The minimum bility occurs in 20% ethanol (2·79 mg of Cs metaperiodate per 5 ml of soln. at 0°). The ee of co-pptn. of Cs during the determination K is a function of the concn. of Cs; as much as g of Cs is co-pptd. with 5 mg of K, despite the bility of the caesium salt. At lower concn. of pptn. is slow and little co-pptn. occurs.
P. D. Parr-Richard

3885. **Determination of radioactive caesium.** I. Handley and C. L. Burros (Anal. Chem. Div., Ridge Nat. Lab., Tenn., U.S.A.). *Anal. m.*, 1959, **31** (3), 332–334.—Caesium is separated, ecessary, from K, NH_4^+ and Rb by pptn. as tungstosilicate, followed by scavenging with $H)_3$ and $BaCO_3$, and finally pptd. with Na phenylboron (**I**). *Procedure*—Add 1 ml of dard Cs carrier to the sample, followed by 5 mg of Fe, Ba, La and Zr carriers. Dilute to 15 ml, add 1 *M* NaOH until just alkaline, then 1 ml of 3 *M* Na_2CO_3. Warm, centrifuge and discard the ppt.; acidify the supernatant liquid, add 5 mg each of Fe, Ba, La and Zr carriers, and repeat the pptn. with NaOH and Na_2CO_3 soln. Make the final supernatant liquid just acid with HCl, cool to 0° and add 4 ml of **I** (4 g of **I** and 1 g of $AlCl_3.6H_2O$ in H_2O, made just alkaline, filtered and made up to 200 ml). Set aside for 10 min., centrifuge and wash the ppt. with H_2O. Dissolve the ppt. in acetone, add 1 ml of *N* HCl and dilute to 10 ml with H_2O. Cool, and repeat the pptn. Dissolve the ppt. in ≃ 1 ml of acetone, add 10 ml of abs. ethanol containing 0·5% of **I**, and cool at 0° for 10 min. Filter on a tared paper and wash the ppt. with ethanol. Dry at 110° and weigh. Distinguish between ^{136}Cs and ^{137}Cs with a multi-channel γ-ray spectrometer. P. D. Parr-Richard

3886. **Amperometric analysis with diethyldithiocarbamate. I. Determination of copper.** Yu. I. Usatenko and F. M. Tulyupa. *Izv. Vyšh. Ucheb. Zavedenii, Khim. i Khim. Tekhnol.*, 1958, (3), 56–60; *Ref. Zhur., Khim.*, 1959, (5), Abstr. No. 15,078.—It is shown that Na diethyldithiocarbamate (**I**) is readily oxidised at the rotating platinum electrode at pH 4·5 to 7 and two waves are obtained at 0·4 and 0·8 V. The diffusion current at 0·7 to 0·9 V is proportional to the concn. of **I**. An amperometric titration of metals that form an insoluble ppt. or complex with **I** is therefore feasible. The amperometric titration of Cu in the presence of EDTA (**II**) (⩾ 0·02 *M* to avoid the development of a residual current) is not affected by Zn, Ni and Fe in 10-, 12- and 17-fold excess, respectively. Lead (60-fold excess) is masked with a Na acetate - EDTA mixture or separated by the chloride method. Aluminium does not interfere, but when a large excess is present it is complexed by means of NaF (ratio of F to Al is 3:1). *Procedure for Cu in aluminium alloys*—Dissolve the alloy (0·5 g) in HCl (1:1) (15 ml) in the cold and then heat. After effervescence has ceased add conc. HNO_3 (1 to 2 ml) and boil for a few minutes until the sample is completely dissolved and the nitrogen oxides are driven off; make the soln. up to 40 to 50 ml with H_2O, cool, neutralise with Na_2CO_3 until no more CO_2 is evolved and make up to 250 ml with H_2O. Take a 10-ml aliquot of this soln., add 3·5% Na acetate soln. (10 ml), satd. NaF soln. (2 ml) and 0·1 *M* **II** (5 ml), make up to 50 ml with H_2O and titrate with 2% **I** soln. The determination takes 30 to 35 min. *Procedure for the determination of Cu in Babbitt metal*—Dissolve the sample (0·5 g) by heating in conc. HNO_3 (15 ml), evaporate to 3 to 5 ml, add 40 to 50 ml of hot H_2O, allow to stand and then filter. Wash the ppt. 3 to 4 times with hot HNO_3 (1:2) and then a few times with hot H_2O. Cool the filtrate, add aq. NH_3 soln. until the hydroxide ppt. begins to appear, carefully add a few drops of HNO_3 until the ppt. is completely dissolved, make up to 250 ml with H_2O, take a 10-ml aliquot and determine the Cu as before, but without the addition of NaF. The removal of Cl^-, NO_3^-, SiO_2 and meta-acids is unnecessary in the analysis of lead-base Babbitt metal. K. R. C.

3887. **Use of organic compounds in precipitation chromatography [of copper and nickel].** K. M. Ol'shanova and N. M. Morozova. *Trudy Moskovsk. Tekhnol. Inst. Myas. i Moloch. Promish.*, 1958, (8), 119–127; *Ref. Zhur., Khim.*, 1959, (7), Abstr.

No. 23,007.—Benzidine and dithio-oxamide are suggested for the determination of Cu, and dimethylglyoxime for Ni. The effects of different types of adsorbents, the relative proportions of the pptg. agent and the carrier, the degree of pulverisation of the carrier, the method of packing the adsorbent, the diam. of the column, the exchange capacity of the adsorbent, the pH of the soln. and the presence of foreign ions on the magnitude of the coloured zone and on the proportionality between the length of the coloured zone and the concn. of the ion being determined were studied. The best chromatograms were obtained on Al_2O_3 and powdered glass, with a ratio of carrier to pptg. agent of 100:1 for dithio-oxamide and dimethylglyoxime and 100:8 for benzidine, and with the carrier well pulverised. At pH > 2 the amount of ppt. depends only on the concn. of the ion being determined; the zone length decreases with an increase in the exchange capacity of the adsorbent. The determination of Cu is affected by the presence of Ni and Co, and the determination of Ni is affected by Cu, Co and polyvalent anions. A method is described for calculating the concn. of the ion being determined from curves of zone vol. (ml) *vs.* dynamic exchange capacity in milliequiv. per g, without constructing calibration curves. K. R. C.

3888. **Gravimetric method for the determination of beryllium in the presence of other elements in alloys and minerals.** T. I. Pirtea and V. Constantinescu (Lab. of Inorg. and Anal. Chem., Univ. C. I. Parhon, Bucharest, Romania). *Z. anal. Chem.*, 1959, **165** (3), 183–188.—The pptn. of Be with $[Co(NH_3)_6]Cl_3$ (**I**) is carried out in the presence of Cu, Cd, Zn, Co, Ni, Fe, Al, Ti, Mn, Ca, Na, K and NH_4^+ by using EDTA as masking agent. *Procedure*—To 5 to 25 ml of a soln. of Be in dil. HCl, containing these cations, are added 0·5 to 1 g of NH_4Cl and 0·5 to 2 g of EDTA. The addition of EDTA is increased for large amounts of Fe. A few drops of 10% aq. NH_3 are added to dissolve any pptd. EDTA. After the addition of 2 to 3 g of powdered ammonium carbonate, Be is pptd. dropwise with conc. **I** soln. until a yellow coloration of the soln. indicates an excess of **I**. The soln. is then diluted to a concn. of 2 to 3% of HCl. The ppt. is set aside for 1·5 to 2 hr., collected in a porcelain crucible of porosity A3 or 10JG3, washed first with 0·2% **I** soln., then 3 or 4 times with 2 to 3 ml of a mixture of 60 ml of ethanol, 40 ml of H_2O and 10 drops of 0·2% **I** soln., then 2 or 3 times with 2 to 3 ml of abs. ethanol and finally 2 or 3 times with 2 to 3 ml of diethyl ether. After drying *in vacuo* for 10 min. it is weighed immediately. The results are tabulated.

B. B. Bauminger

3889. **Chemico-spectrographic method for the analysis of metallic beryllium and beryllium oxide of high purity.** A. G. Karabash, Sh. I. Peizulaev, R. L. Slyusareva and V. M. Lipatova. *Zhur. Anal. Khim.*, 1959, **14** (1), 94–99.—The elements Mg, Ca, Ba, Al, Ti, V, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd, Sn, Pb, Sb, Bi, Ga, In, Tl and Te are determined simultaneously, and Na is determined separately, in high-purity beryllium or beryllium oxide. Dissolve the sample of beryllium (2 g) or beryllium oxide (5·6 g) in 6 *N* HCl (90 ml) and 14 *N* HNO_3 (10 ml), evaporate the soln. till syrupy, and then evaporate with HNO_3 (50, 20 and 20 ml); add conc. aq. NH_3, dry the resulting paste and ignite to oxide at 250° to 300°. Dissolve in glacial acetic acid (100 ml), and evaporate the soln. to dryness to obtain the basic acetate $Be_4O(CH_3COO)_6$, and dissolve this in $CHCl_3$ (100 ml); extract the soln. with 4 *N* HCl (30, 20 and 20 ml) to obtain a soln. containing 85 to 100% of the impurities and about 5% of the Be, evaporate the soln. till syrupy in a weighed quartz dish, treat the syrup with HNO_3 (8, 3 and 3 ml), evaporating after each addition till syrupy, add conc. aq. NH_3, dry the paste and ignite it, first at 300°, then for 30 min. at 500°, and weigh the resulting vitreous BeO to obtain the enrichment factor (original wt. of sample as BeO to wt. of concentrate); this is usually 20 to 25. Carry out a similar procedure with spectroscopically pure $Be(NO_3)_2$ for use as a control. Prepare standards by adding standard soln. of the impurity elements to a soln. of spectroscopically pure $Be(NO_3)_2$, which is then converted into vitreous BeO. Photograph the spectra of the sample containing the concentrated impurities, the BeO derived from the original sample, the controls and the standards on one plate, the spectrogram being used to determine the 24 impurity elements indicated above. For the determination of Na, use a separate series of standards. Construct calibration curves for each impurity. The mean error for all the determinations is $\pm$ 20%; near the sensitivity limits individual errors may reach 50 to 100%, particularly with Cd. C. D. Kopkin

3890. **Determination of magnesium in the presence of large amounts of zinc. A spectrophotometric method.** M. J. Maurice (N.V. Onderzoekingsinst. "Research", A.K.U., Arnhem, Netherlands). *Anal. Chim. Acta*, 1959, **20** (2), 181–186.—The sample soln., which contains up to 400 mg of Zn^{2+} and 40 to 400 μg of Mg^{2+}, is acidified with 3 ml of 4 *N* H_2SO_4 and diluted to 50 ml. Zinc (together with some other metal ions) is first removed by electrolysis at a mercury cathode. The acidity of the soln. is then reduced by passing it through a column of Dowex 3 and, after making it just acid to neutral red, it is extracted with a 5% soln. of 8-hydroxyquinoline in $CHCl_3$ to remove Al^{3+}. Magnesium is then determined spectrophotometrically at 505 mμ by means of 3-hydroxy-4-*o*-hydroxyphenylazonaphth-2′:4′-xylidide (magon) (*cf.* Mann and Yoe, *Anal. Abstr.*, 1957, **4**, 2109). Magnesium is determined by this method in soln. containing added Zn and small amounts of Mn, Fe, Ni, Al, Pb and Ca. H. N. S.

3891. **Determination of magnesium in zirconium by means of ion-exchange chromatography.** S. V. Elinson and M. S. Limonik. *Zavod. Lab.*, 1958, **24** (12), 1434–1436.—The column (40 cm × 1·2 cm) consists of SBS resin containing active $-SO_3H$ groups and retains Mg from an oxalate soln. whilst Zr passes through. To determine Mg in zirconium the sample (1 to 1·5 g) is dissolved in dil. HF (1:5) and the soln. is evaporated to fuming after addition of 1 to 2 ml of conc. H_2SO_4. The residue is dissolved in water and an aliquot is neutralised with aq. NH_3 [pptn. of $Zr(OH)_4$]. After addition of solid oxalic acid until the soln. is clear, the soln. is passed at a rate of 3 to 4 ml per min. through the column which has been treated with 10% HCl soln. and washed free of Cl^- with water. The column is then washed with water to remove Zr, and the Mg is eluted by means of $\leqslant$ 150 ml of 5 to 7% HCl soln. The Mg is determined volumetrically by means of 8-hydroxyquinoline or colorimetrically with Titan yellow (C.I. Direct Yellow 9). Most of the Ca present in the metal can be filtered off as oxalate

efore the use of the column; residual Ca is adsorbed y the column but does not interfere. In the resence of oxalate, Hf, Ti, Fe and Al pass through ie column with the Zr. G. S. Smith

3892. **Preparation and properties of 3:6-dihydroxy-2:4-bis-[NN′-di(carboxymethyl)aminomethyl]fluoran. Utilisation for the ultra-micro determination of calcium.** D. F. H. Wallach, O. M. Surgenor, J. Soderberg and E. Delano Dept. of Biol. Chem., Harvard Med. Sch., Boston, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 456–460.—The reagent is prepared by condensation of iminodiacetic acid, formaldehyde and fluorescein. Details are given of its preparation and of its use as a fluorescent indicator in the complexometric titration of calcium. H. F. W. Kirkpatrick

3893. **Estimation of radioactive calcium-45 by liquid scintillation counting.** L. Lutwak (Metabolic Diseases Branch, Nat. Inst. of Health, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 340–343.—Calcium-45 is extracted from biological material and is then concentrated and dissolved in ethanol to which is added 0·4% diphenyloxazole in toluene. It is then determined by a liquid scintillation counting method with a counting efficiency of about 65% and a coeff. of variation of 7·4%. K. A. Proctor

3894. **Micro-titration of calcium to visible end-point in presence of magnesium. Sodium (ethylenediamine)tetra-acetate as titrant.** S. J. Socolar and I. Salach (Coll. of Physiol., Univ., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 473–474.—As little as 0·2 μg of Ca can be determined by the method described. Calcium added to biological samples gave 100·8% recovery. Interference by Fe and Cu is suppressed with CN^-, but a 100-fold molar excess of Mg does not interfere. *Procedure*—Pipette the sample (0·5 ml), 0·3 ml of cyanide mixture (1 *N* in NaOH, 0·7 *N* in KCN) and calcein (5 ml, from 1 drop of 2% soln. diluted to 25 ml) into a white porcelain crucible. Prepare a blank similarly and illuminate both crucibles with concentrated light from a tungsten source passed through a triple filter (Wratten No. 78AA); the blank will appear pink and the sample greenish yellow. Titrate with 0·002 *M* EDTA (disodium salt) from a micro-burette until the sample matches the blank. Crucibles and glassware (except for silicone-coated pipettes) should be soaked before use in 2 *N* HNO_3 and rinsed with metal-free H_2O.
P. D. Parr-Richard

3895. **Calcium carbide.** British Standards Institution (2 Park St., London, W.1). B.S. 642:1951. Amendment No. 2, 14.5.1959.—A correction is made in the method for the determination of arsenic compounds.

3896. **Determination of strontium in silicates and carbonates in a flame photometer with a liquid colour-filter.** G. I. Ksandopulo and D. P. Shcherbov Central Lab., South Kazakhstan Geol. Dept.). *Zavod. Lab.*, 1958, **24** (12), 1432–1434.—The strontium band at 640 to 690 mμ with a 1-cm thickness of a 1% aq. soln. of Rhodamine S (C.I. Basic Red 11) as a colour filter is used. Compensation for the Ba and Ca present is made on the basis of measurements at 470 to 540 mμ with a 5-cm thickness of 100% aq. $Cu(NO_3)_2$ soln. or 40% aq. $CaCl_2$ soln. G. S. Smith

3897. **Determination of barium and sulphate with an EEL flame photometer.** D. C. Cullum and D. B. Thomas (Colgate-Palmolive Ltd., 371, Ordsall Lane, Manchester, England). *Analyst*, 1959, **84**, 113–116.—The method was devised for the determination of SO_4^{2-} in detergent powders. The sample (2 g) in 100 ml of water is acidified with HCl and extracted with diethyl ether, the extract is rejected and the aq. layer is diluted to 250 ml. A 5-ml aliquot is centrifuged with 5 ml of 1% $BaCl_2$ soln., the supernatant liquid is discarded, and the ppt. is washed once with water in the same manner. It is then suspended in a 1% starch soln., the suspension is adjusted to 10 ml and is analysed in an EEL flame photometer with a Wratten No. 65A or mercury monochromat No. 77 filter, the instrument being set to zero with 1% starch soln. A standard suspension is prepared by treating a mixture of 1 ml of a standard SO_4^{2-} soln. (containing 1% of SO_4^{2-}), 4 ml of water and 5 ml of $BaCl_2$ soln. in the same manner as the sample. The final suspension (≡ 1000 p.p.m. of SO_4^{2-}) is used to set the instrument to full-scale deflection. The method is applicable to the determination of Ba but, since the Ba flame is sensitive to anionic interference, the scale reading will vary with changes in the anion. A. O. Jones

3898. **Rapid micro-determination of mercury by spectrophotometric flame combustion.** O. Lindström (Lab. Dept., AB Casco, Stockholm, Sweden). *Anal. Chem.*, 1959, **31** (3), 461–467.—Millimicrogram quantities of Hg in urine and aq. soln. can be determined by this method, in which the sample is burned in an atomiser burner and water vapour and salt mist are removed by condensers and cotton filters. The mercury content of the dried gas is then determined by means of a mercury vapour meter. The coeff. of variation on dilute aq. soln. is ± 1% and the limit of detection is 10^{-9} g of Hg. The method can be applied to disinfectants, paper and pulp products, and seed-treating materials. K. A. Proctor

3899. **Separation of zinc from indium by electrolysis with a mercury electrode.** M. G. Sayun, P. P. Tsyb and K. Kh. Latkher (Mining Metallurgy Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1958, **24** (12), 1436–1439.—Anodic, but not cathodic, decomposition of an amalgam containing In and Zn can be used to separate the metals. A mercury-mercurous sulphate half-cell is used as a reference electrode. The sample (10 to 20 g) of indium is dissolved in 10 to 15 ml of mercury in an electrolysis cell containing a spiral platinum electrode and a connection to the half-cell. After addition of 50 ml of *N* H_2SO_4, the anode potential is measured. If the value is approx. that of the normal potential for Zn (— 0·77 to — 0·55 V) the soln. is heated to 80° and the amalgam is decomposed electrolytically, the current being regulated so that the anode potential does not become more positive than — 0·48 V. When the current is near zero, the decomposition is continued without application of external e.m.f., and when the anode potential attains the value required for oxidation of In (≃ — 0·4 V) the decomposition is continued for a further 60 min. Transference of Zn from amalgam to soln. is practically complete, but ≃ 10 to 15 mg of In also dissolves. The aq. soln. is separated and Zn is determined polarographically. If the amount of Zn is so small that the potential of the amalgam corresponds to that for oxidation of In, the

decomposition is carried out for 60 min. at 80° without application of external e.m.f.

G. S. Smith

3900. **Preparation of new solution standards of radium.** W. B. Mann, L. L. Stockmann, W. J. Youden, A. Schwebel, P. A. Mullen and S. B. Garfinkel (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand.*, 1959, **62** (1), 21–26.—The preparation is described of (*i*) a master soln. containing $2{\cdot}001 \times 10^{-6}$ g of ^{226}Ra per ml of carrier soln. (0·2% $BaCl_2.2H_2O$ + 5% HCl), and (*ii*) two soln. containing, respectively, 10^{-9} and 10^{-11} g of Ra per 100 ml, obtained by dilution of the master soln. Comparison of these soln. with the 1940 and 1947 standards (10^{-9} and 10^{-11} g), by radon analysis, indicates an error of $\simeq +2{\cdot}6\%$ of Ra in the 1940 standards. The error is ascribed to the dilution procedure.

W. J. Baker

3901. **Determination of boron concentration by exposure to slow neutrons.** L. I. Shmonin, V. V. Cherdyntsev and D. I. Tartovskiĭ. *Uch. Zap. Kazakhsk. Univ.*, 1957, **30**, 7–11; *Ref. Zhur., Khim.*, 1959, (7), Abstr. No. 23,070.—The method is based on the greater capacity of B (100 times that of other elements) to absorb slow neutrons with the liberation of α-particles and ^7Li nuclei (in the reaction $^{10}B + n = He + {}^7Li$) the quantities of which are practically proportional to the concn. of B in the test sample. A layer (3 to 4 mg per sq. cm) of finely ground test material is exposed to neutrons from a Ra - Be source ($\simeq$ 0·15 C) which are slowed down by a paraffin-wax block (7 cm thick), and the number of α-particles and ^7Li nuclei emitted by the sample in unit time is measured by means of a counter with an impulse ionisation chamber. The concn. of B is found by means of a calibration curve constructed from standards of $CaCO_3$ with known quantities of H_3BO_3 added. When the natural radioactivity of the test samples exceeds 0·5 impulse per min., a reading is also taken before exposure to neutrons. The mean error for concn. of B of $\not<$ 1% is $\pm$ 10%; the determination takes $\simeq$ 0·5 hr. If the concn. of B is reduced to 0·1%, the mean error is raised to $\pm$ 30%, but the determination is shortened by a few hours.

K. R. C.

3902. **Mass-spectrometric analysis of the isotopic composition of boron.** G. A. Semenov and Yu. A. Zonov (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), 137–138.—The isotopic composition of B is determined by evaporation of B_2O_3 or H_3BO_3 at 1000° to 1200° from a platinum ribbon. From B_2O_3 the ions $B_2O_3^+$, $B_2O_2^+$, BO^+ and B^+ were observed, in the ratio 19 : 3·1 : 2·2 : 1; $B_2O_2^+$ and B^+ were used for determining the isotopic composition. The results were checked by using BF_3 as source.

C. D. Kopkin

3903. **Determination of aluminium [in ceramic materials] with Complexone III.** I. A. Voinovitch, J. Debras, C. Yelatchich and Z. Zalessky (Soc. Française de Céram., Paris). *Ind. Céram.*, 1958, (501), 291–295.—After decomposition and dissolution of the sample, the SiO_2 is made insol. by fuming with H_2SO_4 and the Al, Ti and Fe in an aliquot ($\equiv$ 20 to 50 mg of Al_2O_3) are complexed, at $\simeq$ pH 4·5 and 100°, with an excess of 0·05 *M* EDTA (disodium salt). The excess is then back-titrated with 0·05 *M* $ZnCl_2$ in the presence of ethanolic dithizone (0·025%, w/v) as indicator, the end-point being a sharp change in colour from greenish violet to red. In calculating the content of Al, allowance should be made for the percentages of Fe and Ti (determined concurrently by standard methods on separate aliquots). Neither Ca nor Mg interferes, but Pb, W and other heavy metals should be absent. The method is accurate (max. error 0·5%) and rapid (7 analyses can be completed in $\simeq$ 2 hr.); it is strongly recommended for the routine analysis of clays and similar alumino-silicates.

W. J. Baker

3904. **Spectrophotometric determination of aluminium with quinizarin-2-sulphonic acid [1 : 4-dihydroxyanthraquinone-2-sulphonic acid] (sodium salt).** E. G. Owens, II, and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 384–387.—The violet complex of Al^{3+} with Na quinizarinsulphonate (**I**) is applicable to spot testing and to the quant. determination of Al. Interfering ions (except Be^{2+}, Sc^{2+}, Th^{4+}, Ti^{4+}, Y^{3+}, Zr^{3+}, F^-, PO_4^{3-} and those of rare-earth metals) are removed at a mercury cathode. *Procedure*—After removal of Fe, etc., pipette an aliquot (30 to 70 μg of Al) containing 2 to 15 ml of conc. H_2SO_4 per 100 ml into a silica dish. Evaporate gently to fuming, then take to dryness. Cool, add 10 drops of conc. HCl and 5 drops of conc. HNO_3, and H_2O to 5 ml. Evaporate, dissolve the moist residue in 5 ml of abs. methanol and transfer with methanol to a 50-ml flask. Add 10 ml of reagent (0·16 g of dry **I** in abs. methanol, made up to 500 ml and stored in polyethylene), and methanol to 50 ml; the final pH should be 0·3 to 0·5. Read the extinction after 1 hr. at 560 mμ against a reagent blank. Beer's law is obeyed up to 1·7 p.p.m. of Al; the sensitivity is 1 part of Al in 5×10^8 parts of soln.

P. D. Parr-Richard

3905. **Comparison of methods of determining organic substances in aluminate solutions.** D. V. Bezuglyĭ and I. A. Petrusevich (V. I. Lenin Kharkhov Polytech. Inst.). *Zavod. Lab.*, 1958, **24** (12), 1448–1449.—Various published methods for determining organic matter (humates, starch, etc.) in aluminate soln. are found to be unsatisfactory and an improved method is described. The sample (5 ml) is first neutralised and then treated further with H_2SO_4 to give an acid concn. of 40%. After addition of 2 ml of 0·5 *N* $K_2Cr_2O_7$, the soln. is heated under a reflux condenser on a boiling-water bath for 2 hr., after which it is cooled and mixed with 20 ml of 0·1 *N* ferrous soln., and the excess is titrated with 0·1 *N* $KMnO_4$.

G. S. Smith

3906. **The polarographic properties of gallium and indium.** A. I. Zelyanskaya, N. V. Bausova and L. Ya. Kukalo. *Trudy Inst. Metallurg. Ural'sk. Fil. Acad. Nauk, SSSR*, 1958, (2), 263–274; *Ref. Zhur., Khim.*, 1959, (6), Abstr. No. 19,101.—It is shown that, with a basal electrolyte of 0·1 *M* Na salicylate and 0·1 *M* NaCl at pH 2·5 to 3·8, Ga forms a satisfactory polarogram with $E_{\frac{1}{2}} = -0{\cdot}988$ *vs.* the S.C.E.; the introduction of gelatin into the test soln. markedly diminishes the height of the Ga wave and affects its shape. In the polarography of In, it is shown that, with an increase in concn. of HCl in soln., $E_{\frac{1}{2}}$ is shifted to more negative values. The addition of gelatin to the test soln. diminishes the height of the diffusion wave. With 0·1 *N* to 0·4 *N* HCl, complexes of the $InCl_2^+$ type are formed with $K_n = 5{\cdot}27 \times 10^{-7}$ but, with 0·

5 *N* HCl, complexes of the $InCl_4^-$ type are ned, with $K_n = 3{\cdot}63 \times 10^{-8}$. In 3 *N* HCl in presence of 0·01% of gelatin the direct pro- tionality between the diffusion current and the cn. of In in the soln. is maintained. K. R. C.

907. **Polarographic determination of indium.** T. Nizhnik and I. S. Chaus (Inst. of Gen. and rg. Chem., Acad. of Sci., UkrSSR, Kiev). *Zhur. il. Khim.*, 1959, **14** (1), 37–40.—To determine in industrial concentrates and products, de- ıpose the sample (1 to 3 g) by heating with SO_4 and HNO_3 to white fumes, cool, add hot er, filter if necessary, add to the filtrate H_2SO_4 equiv. sulphate) to a concn. $\not< 20\%$, treat with amalgam for $\simeq 45$ min., filter if necessary, about 10% by wt. of an alkali chloride (*e.g.*, Cl) and polarograph. No interference is caused Cd, Sn, Tl, Cu, As, Sb, Bi, Se, Te, Fe, V, Cr Ti. C. D. KOPKIN

908. **Reactions of ions of tervalent thallium n iodine: the iodine complexes of thallium.** A. I. ev and V. G. Tiptsova. *Nauch. Dokl. Vyssh. ol. Khim. i Khim. Tekhnol.*, 1958, (3), 486–490; . *Zhur., Khim.*, 1959, (6), Abstr. No. 19,103.— s shown that, on the addition of KI to $Tl_2(SO_4)_3$ ı., the following reactions take place: $Tl^{3+} +$ $\rightarrow Tl^+ + I_2$, $I_2 + I^- \rightleftarrows I_3^-$, and $Tl^+ + I^- +$ $\rightleftarrows TlI_4^-$. The reaction of Tl^{3+} with I^- proceeds ctly stoicheiometrically and is expressed by the equation for concn. of Tl of 10^{-4} to 10^{-5} *M*, can therefore be used for the titrimetric deter- ation of Tl. In the presence of Cl^-, Br^- or ate, the stoicheiometric relationship is disturbed ı concn. of Tl of 10^{-3} to 10^{-2} *M* because of pptn. of TlI as a consequence of the formation hloride and bromide complexes of Tl^{3+} and formation of I_2Br^-. *Volumetric determination*— 0·2% starch soln. (1 to 2 ml) to a soln. of $SO_4)_3$ or $Tl(NO_3)_3$ (10 to 20 ml) containing ·2 mg of Tl and made 0·1 *N* with acid; titrate 0·001 or 0·002 *M* KI. The determination is affected by Zn, Fe or Cu; Cd and Hg^{2+} fere. *Spectrophotometric determination*—The ation of TlI_4^-, which has a max. absorption at mμ, is used for the determination of 1 to 15 μg Tl in the presence of a 3000-fold excess of KI prevent dissociation of TlI_4^-). Add 10% KI (5 ml) to the test soln. containing 0·05 to mg of Tl; make up to 25 ml with H_2O and sure the extinction at 395 mμ in a 10-mm The determination is affected by elements ing complexes with I^- (*e.g.*, Bi, Cu, Hg, Ag Pb) and by oxidising agents (Fe^{3+}, Sb^{5+} and); HCl and H_2SO_4 do not interfere. K. R. C.

909. **Use of vaporisation analyses for the ration of trace substances. II. The identifica- and determination of traces of thallium.** W. nann and K.-H. Neeb (Saarstrasse 21, Mainz, many). *Z. anal. Chem.*, 1959, **165** (4), 251– —The method is based on the relatively high ur pressure of Tl and its compounds. The sublimed in a stream of H or O and is con- ed on the quartz or aluminium cap of a cold and measured spectrographically. The cap ed directly as the electrode. The temp. of mation and the carrier gas used depend on ature of the sample; O is used when other volatile metals are present; the oxides ed do not interfere with the determination. method is applicable to soln.; the Tl is pptd. hen sublimed, or is extracted with ether and concentrated by evaporation, then pptd. from aq. soln. with NaOH, with $MnSO_4$ as carrier. A max. error of 30% is obtained in the concn. range 2 to 100×10^{-8} g of Tl. The spectral lines used depend on the concn. of Tl, and methods for various samples are given. G. P. COOK

3910. **Reduction of thallium(III) to thallium(I) with metallic reductors.** R. F. Rolf and E. Leininger (Kedzie Chem. Lab., Michigan State Univ., East Lansing, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 425–426.—The reducing action of metallic bismuth, cadmium and silver on dil. H_2SO_4 soln. of Tl^{III} was examined; the reaction was quant. in all cases. *Procedure*—Pass a 50 to 100-ml aliquot of thallium soln. containing 0·1 to 1·0 *N* H_2SO_4 through a 20-cm column of metallic reducer at about 20 ml per min. and wash the column with 0·1 *N* H_2SO_4 (3 × 50 ml). Add 40 ml of 6 *N* HCl to the soln. and washings, and titrate Tl^I with 0·1 *N* $KBrO_3$ potentiometrically. Blank corrections are small and for 5 determinations on 32-mg samples (cadmium column), the standard deviation was ± 0·22 mg. Bismuth and cadmium are more convenient as reducers than silver, since no ppt. is formed with HCl. Lead, aluminium and zinc and amalgams of zinc, cadmium, lead and bismuth were unsatisfactory. Nickel gave quant. results but high blanks. P. D. PARR-RICHARD

3911. **Determination of thallium by high-frequency titration with sodium tetraphenylboron.** A. R. Veĭs and A. F. Ievin'sh (Latvian State Univ., Riga). *Zhur. Anal. Khim.*, 1959, **14** (1), 143–144.—Thallium is determined by high-frequency titration of Tl_2SO_4 with 0·2 *M* Na tetraphenylboron; by using 0·5 to 2·0 ml of 0·1 *N* Tl_2SO_4 in 25 ml, no interference was caused by NaCl, $ZnSO_4$, $CdCl_2$, $Cu(NO_3)_2$, Na_2CO_3, H_2SO_4 or acetic acid. The max. error was 1%. C. D. KOPKIN

3912. **Dichrometric determination of thallium(I).** K. Bhaskara Rao (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **165** (3), 193–195 (in English).—It is shown that for the volumetric determination of Tl^+ with $K_2Cr_2O_7$ it is expedient to use an organic layer to detect the end-point with ICl. *Procedure*—The Tl^+ soln. is placed in a 250-ml iodine flask, followed by 5 ml of 0·02 *M* ICl, sufficient conc. HCl to keep the over-all acidity at $\not< 7{\cdot}5$ *N* at the end-point, and water to a total vol. of 50 ml. The soln. is allowed to cool to room temp., 5 ml of CCl_4 is added and titration with 0·05 *N* $K_2Cr_2O_7$, with thorough shaking, is carried out until the purple colour of the organic layer is discharged.

B. B. BAUMINGER

3913. **Photometric determination of thallium.** H. Zimmer (Mehringdamm 53, Berlin SW 61, Germany). *Z. anal. Chem.*, 1959, **165** (4), 268–271.—Thallium is ppt. as insol. Tl_2CrO_4, which is filtered off, washed and dissolved in 6 *N* HCl. The CrO_4^{2-} are determined photometrically with diphenylcarbazide. The concn. range is 0·7 to 1·2 mg in the presence of $\simeq$ 500 mg of Li, K, Na, Cs or Rb, and of $\simeq$ 15 mg of Zn^{2+}, Cd^{2+} or Co^{2+}; up to 20 mg of AsO_4^{3-} or $Sb_2O_7^{4-}$ does not interfere; Cr^{3+}, Al^{3+} or Be^{2+} (2 mg) do not interfere after addition of sulphosalicylic acid. These amounts must not be exceeded, as the solubility of Tl_2CrO_4 increases with high salt concn. The mean error is $\not> \pm 0{\cdot}01$ mg of Tl in the presence of these ions. G. P. COOK

3914. **Separation of tracer quantities of lanthanides from plutonium using calcium fluoride.** P. M. Aron. *Atomnaya Energiya*, 1958, **5** (2), 183–185; *Ref. Zhur., Khim.*, 1959, (7), Abstr. No. 22,985.—*Procedure*—Add conc. HNO_3 satd. with silver peroxynitrate ($Ag_7O_8.NO_3$) and $\simeq$ 40% HF (150 mg) to a soln. (0·5 to 1·0 ml) containing tracer quantities of lanthanides and ^{239}Pu (0·34 mg), make the soln. *N* with HNO_3 and introduce Ca^{2+} (6 mg); $Ag_7O_8.NO_3$ oxidises Pu^{4+} to Pu^{6+}. The pptd. CaF_2 contains all the lanthanides present (confirmed by the β-activity of ^{151}Eu, ^{154}Eu, ^{144}Ce and ^{144}Pr) and $\simeq$ 3% of Pu. Re-precipitate to obtain a ppt. containing $\simeq$ 0·08% of Pu. Since the acidity of the soln. and the continued ageing of the ppt. have no effect on the completeness of lanthanide recovery, the evidence points to the formation of a solid soln. of lanthanides in the CaF_2 ppt. The method described can be used for the mass-spectrometric analysis of lanthanides and for their recovery from Pu without carriers.

K. R. C.

3915. **New photometric determination of cerium with methylene blue.** Hidehiro Gotô and Yachiyo Kakita (Inst. Iron, Steel and other Metals, Tohoku Univ., Katabira-cho, Sendai). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (12), 1524–1528.—The coloured substance formed from Ce^{IV} and methylene blue (**I**) in a weakly basic soln. (Passerini, *Gazz. Chim. Ital.*, 1935, **65**, 824; Reed, *Analyst*, 1938, **63**, 338) is extracted with benzene, $CHCl_3$, CCl_4, amyl acetate or ethyl acetate to give a purple soln. (max. absorption, 510 mμ in benzene and CCl_4, 525 mμ in $CHCl_3$). Addition of 2 ml of 2 *N* NaOH to a neutral soln. of the reactants is recommended. The working curve is rectilinear for $<$ 40 μg of Ce per 5 ml of benzene. Ammonium ions, Cl^-, NO_3^- and ClO_4^- decrease the intensity, whereas PO_4^{3-}, F^-, oxalate, tartrate, citrate, acetate and EDTA mask the reaction. Bivalent Mn gives a similar coloration. Mixed oxides of rare-earth elements are dissolved in 6 *N* H_2SO_4 and a portion is diluted to produce $\simeq$ 15 ml of *N* H_2SO_4 soln. The product is treated with $AgNO_3$ ($\simeq$ 150 μg) and $K_2S_2O_8$ (0·2 g) at 50°, cooled, mixed with **I** soln. (0·01%, 1 ml) and benzene (5 ml) and neutralised. The soln. is shaken with 2 *N* NaOH (2 ml), for 1 min., and the extinction of the organic layer is measured.

K. Saito

3916. **Colour reactions for the separation of lanthanum from cerium.** F. S. Frum. *Trudy Khim. i Khim. Tekhnol.*, 1958, (1), 132–133; *Ref. Zhur., Khim.*, 1959, (6), Abstr. No. 19,106.—It is shown that Acid Monochrome Bordeaux S (**I**) can be used as a reagent for detecting La^{3+}, and gallein (**II**) can be used for detecting Ce^{3+}. *Procedure for* La^{3+}—To the test soln. (1 ml) add buffer soln. (0·2 ml) at pH 9 to 9·4 and a 0·2% aq. soln. of **I**; an orange colour appears if La^{3+} are present (a violet colour is obtained in the control). The detectable minimum of La is 1·5 μg per ml; the limiting dilution is 1 in 6·6 $\times$ 10^5; Al, Be, Ca, Ti, Th, Fe^{3+}, U^{4+} and salts of Ce^{3+} and Ce^{4+} do not interfere. **I** can also be used for the colorimetric determination of La; the error is $\gtrsim$ 10%. *Procedure for* Ce^{3+}—To the test soln. (1 ml) add a 0·2% ethanolic soln. of **II** (2 drops); in the presence of Ce^{3+} a blue or violet colour is obtained (a pink - violet or pink colour is obtained in the control). The detectable minimum of Ce is 1·2 μg per ml; the limiting dilution is 1 in 8 $\times$ 10
Nd, Pr and PO_4^{3-} do not interfere; salts of Ce
give a yellow colour with **II**, and a ppt. is sometim
obtained. With **II**, Ce^{3+} can be detected in th
presence of a 200-fold excess of La. **II** can also
used for the quant. determination of Ce; the err
of determination is $\gtrsim$ 10%. K. R. C.

3917. **Indirect titrimetric determination of prase
dymium in admixture with other rare-earth elemen
of the cerium group.** M. N. Ambrozhiĭ and A. M
Gol'tsev. *Nauch. Dokl. Vŷssh. Shkol. Khim.
Khim. Tekhnol.*, 1958, (3), 491–494; *Ref. Zhur
Khim.*, 1959, (6), Abstr. No. 19,107.—A metho
was developed for the detection of Pr in a mixtur
of oxides of La, Nd and Sm [after removal of C
as $Ce(NO_3)_4.2NH_4NO_3$] based on the selectiv
formation of PrO_2 on calcination of a mixture
salts of the rare-earth elements and on the subs
quent oxidation of Mn^{2+} to MnO_4^- by PrO_2
acid soln. *Procedure for the detection of Pr*
Evaporate the test soln. to dryness, ignite for 0
to 1 min. at 600° to 700°, cool, add 1 to 2 drops
$MnSO_4$ soln. (50 mg per ml) and H_2SO_4 (1:3) an
stir; the red - violet colour of MnO_4^- is obtaine
immediately in the presence of Pr. The detectab
minimum is 10 μg of Pr; the limiting dilution
1 in 1 $\times$ 10^6. *Determination of Pr in a mixture
rare-earth-element oxides*—Dissolve the mixture b
heating in HNO_3 (sp. gr. 1·4), evaporate to on
third of the vol., add an excess of NH_4NO_3 soln
evaporate at 60° until crystals of $Ce(NO_3)_4.2NH_4NO$
appear, cool and filter off the ppt. Evaporate th
filtrate to dryness on a water bath, heat the residu
in a muffle-furnace to 700°, ignite for 2 hr. an
weigh. Treat an amount of the calcined oxide
containing $\simeq$ 20 mg of Pr with 4 ml of MnSO
soln. (50 mg per ml) and 12 ml of H_2SO_4 (1:3
and titrate the MnO_4^- formed with 0·1 *N* oxali
acid. With a Pr content of $>$ 15%, use 20 mg
the oxides; the error of determination is $\pm$ 0·2%
with a Pr content of $<$ 15%, use 100 mg; th
error is $\pm$ 1%. It is shown that on ignitin
$Pr_2(NO_3)_3$, 51·4% of PrO_2 is formed and on ignitin
Pr oxalate, 34·5% of PrO_2 is formed, which corr
sponds to the formation of $Pr_2O_3.2PrO_2$ an
$Pr_2O_3.PrO_2$, respectively. K. R. C.

3918. **Separation of rare earths. VII. Th
system praseodymium oxide - neodymium oxid
Determination of available oxygen.** L. Wolf,
Wolff and H. Bärnighausen (Karl Marx Univ
Leipzig, Germany). *J. prakt. Chem.*, 1958,
259–265 (in German).—An apparatus is describe
which permits the gas-volumetric determination
the available oxygen in the mixed oxides of Pr a
Nd. The principle underlying this determinati
is the ability of PrO_2 to be reduced in dil. HNO_3
Pr^{3+} with the evolution of O. Since the Nd prese
is already in the tervalent form, while the Pr is
the quadrivalent form, this method yields dire
information on the Pr content of the mixed oxid
The results obtained on 11 synthetic mixtures
the two oxides confirm the usefulness of this meth

Chem. Abstr.

3919. **Sensitive measuring methods for rad
carbon.** G. Krakau and H. Schneider (Uni
Giessen, Germany). *Atomkernenergie*, 1958,
512–522 (in German).—Application of radio-carb
(^{14}C) for dating or tracer investigations requi
sensitive detection methods. Geiger - Mül
counters, scintillation counters with liquid or so

sphors, and photographic emulsions find applica- The different methods are discussed and pared. A new method is described suitable tracer investigations, in which a phosphor is for sensitive measurement of gaseous $^{14}CO_2$. lly, the different data of the most important ıods are compared. NUCL. SCI. ABSTR.

920. **Gravimetric method for the determination ilica.** H. Bennett (Brit. Ceram. Res. Assoc., e-on-Trent, England). *J. Soc. Glass Tech.*, **43**, 59T–61T.—The quinoline molybdosilicate ıod (*cf.* Bennett *et al.*, *Trans. Brit. Ceram.* , 1958, **57**, 1) is adapted for glass. The ple is fused with NaOH, the melt is dissolved ɔiling water in a nickel beaker and the suspension iluted to ≃ 175 ml, poured into an excess of :. HCl with stirring, cooled and made up to ml. An aliquot, containing ≃ 0·035 g of SiO_2, nade strongly alkaline with NaOH pellets, a slightly acid with HCl. Ammonium molybdate ıdded and after a few minutes the soln. is made ngly acid with HCl and aq. quinoline soln. ıdded from a burette with stirring. The cream- ured ppt. is coagulated by warming to 80°, ed to < 20°, filtered on a G4 sintered-glass ch crucible, washed with quinoline soln., dried 2 hr. at 150° and weighed. J. A. SUGDEN

921. **Micro-methods for the determination of a.** J. González Carreró and O. Carballido allo (Univ., Santiago, Spain). *An. Real Soc. Fís. Quím., B*, 1958, **54**, 191–208.—Existing ıods were studied to determine the effects of ables on the determination of the active and silica content of soln. Methods for SiO_2 rmination based on the formation of molybdo- c acid which follow Beer's law up to concn. 0 μg of SiO_2 per ml are inaccurate if the test contains HCl < 0·05 *N* or > 0·2 *N*. Adjust pH of a test soln. to 2 to 6, add HCl to 0·1 *N*, add 5 ml of 10% $(NH_4)_2MoO_4$ soln. After ing, add 2·5 *N* HCl, then water to 25 ml. - 20 min., read the soln. in a spectrophotometer 430 mμ, this being the extinction max. for bdosilicic acid. The SiO_2 content of Na_2SiO_4 freshly prepared from SiO_2 and Na_2CO_3 by n, was compared with common water-glass ites (**II**) by gravimetric and colorimetric minations. The SiO_2 content of **I** was found the same by either method, but lower results obtained with **II** by the colorimetric method. the amount of active SiO_2 is less than the and varies according to the age of the sample. merised SiO_2 is in equilibrium with the mer in **II**. By using the colorimetric method, SiO_2 content of **I** remains at a constant level ss independent of the pH of the test sample a period of 2 months. In **II**, the active SiO_2 nt increases with the age of the sample and he pH. The disagreement of previous workers on s SiO_2 content is attributed to this phenomenon. nic materials interfere with the molybdosilicate formation by reducing MoO_4^- to blue odenum oxides. Quartz can be assayed ately only if NaOH is used as a flux and the is carried out for at least 5 min. If Na_2CO_3 od, the fusion must be carried out at 1000° hr. The method of Duval, in which SiO_2 verted into the molybdosilicate and pptd. as examine (**III**) salt, then dried at 800° or ed at 600° and weighed, does not give accurate s because **III** precipitates some molybdate as well. The method is not recommended. Methods involving urea, oxine or other organic precipitants are unreliable unless rigorous precautions are taken. CHEM. ABSTR.

3922. **Rapid method for complete analysis of silicon carbide.** T. Ya. Kosolapova and E. E. Kotlyar (Inst. of Metal Ceramics and Special Alloys, Acad. Sci. UkrSSR). *Zavod. Lab.*, 1958, **24** (12), 1442–1443.—The finely ground (270 mesh) sample (0·4 g) is heated at 800° to 850° to const. wt. (20 to 40 min.) to give the content of free C from the loss in wt. The residue is treated with HNO_3, HF and H_2SO_4 and ignited to give the content of free SiO_2 plus free Si. The residue is then treated with 30 to 40 ml of dil. HCl (1 : 1) and Fe is determined in the filtrate. The insoluble matter is ignited to const. wt. at 800° to 850° to give the content of SiC. To determine free Si, the sample (0·5 to 1 g) is heated for 45 to 60 min. with 60 to 70 ml of 1% NaOH soln., and the Si in the filtrate is determined photometrically by means of the yellow molybdosilicate complex. G. S. SMITH

3923. **Determination of nitrogen in silicon nitride.** Kiichi Narita (Development Dept., Kobe Seiko Co., Nada-ku). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (12), 1564–1566.—Silicon nitride, Si_3N_4, is not decomposed with conc. H_2SO_4, HF, $HClO_4$ or a mixture of $CuSO_4$, K_2SO_4 and H_2SO_4, but is readily fused with NaOH (> 50 times the wt. of the Si_3N_4) at 500° to 600° to form NH_3. Nitrogen in all forms in iron and steel can be determined by this method. *Procedure*—Place the sample (0·1 g) and NaOH (5 g) in a nickel crucible, and heat in an iron or nickel vessel in a current of A (80 to 100 ml per min.). Allow the NH_3 to pass into 0·1 *N* H_2SO_4 (40 ml), and back-titrate with NaOH soln. or measure the colour by the Nessler method (for < 0·5% of N). K. SAITO

3924. **Silicate analysis. A review.** R. C. Chirnside (Gen. Electric Co., Wembley, England). *J. Soc. Glass Tech.*, 1959, **43**, 5T–29T.—An historical and critical review of classical and modern methods concludes with a summary of present and future prospects. (27 references.) J. A. SUGDEN

3925. **Volumetric analysis of stannous and total tin in acid-soluble tin compounds.** J. D. Donaldson and W. Moser (Chem. Dept., Univ. of Aberdeen). *Analyst*, 1959, **84**, 10–15.—In the apparatus described, the sample (≡ ≃ 0·15 g of Sn^{2+}) is placed in a reaction vessel in an atmosphere of CO_2, and de-aerated HCl (2 *N*) is then raised to b.p. and by means of its own vapour pressure is injected into the reaction vessel, which is heated on a water bath until the sample has dissolved. Ferric chloride is then injected into the reaction vessel in the same manner. The reaction mixture is boiled for 10 min., then cooled, and, after addition of H_3PO_4 to decolorise the Fe complex, it is titrated with 0·1 *N* $Ce(SO_4)_2$ with 1 : 10-phenanthroline - ferrous sulphate soln. as indicator. For total Sn, the sample is dissolved in HCl in the reaction vessel, pure aluminium (≃ 0·2 g) is added and CO_2 is passed through the liquid. After ≃ 30 min. the excess of aluminium is dissolved by heating, conc. HCl is injected into the reaction vessel to dissolve the deposited Sn, and the total Sn is determined as Sn^{2+}, as before. A. O. JONES

3926. **Determination of tin in inorganic and organic compounds and mixtures.** M. Farnsworth and J. Pekola (Res. Lab., Metal and Thermit Corp., Rahway, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 410–414.—In the volumetric determination of Sn by titration with KIO_3 soln., the preliminary reduction by nickel is only satisfactory for the determination of total Sn; nickel plus iron should be used for Sn^{II}. Interference by Cu is eliminated by substituting H_3PO_2 for nickel plus iron, adding $HgCl_2$ as catalyst and then KSCN.

P. D. PARR-RICHARD

3927. **Field method for determination of lead fume.** B. E. Dixon and P. Metson (Dept. of the Gov. Chemist, Clement's Inn Passage, London). *Analyst*, 1959, **84**, 46–50.—Test papers (2·5 in. × 1 in.) are dipped to a depth of 1 in. in a 0·2% (w/v) soln. of tetrahydroxy-*p*-benzoquinone in ethanol or methanol, then dried. Through these, mounted in a holder, air containing lead fume is drawn at 3 to 5 litres per min. The exposed side of the paper is sprayed with a mixture of Pb-free glacial acetic acid and acetone (1 + 1) and the uniform purple stain is compared with standard stains. These are prepared from a source of lead fume, and after development of the colour the amount of Pb in an aliquot area of the paper is determined by the dithizone method. The amount of Pb present on a circle of paper 1 cm in diam. is then calculated. Since the stains are not permanent, they may be copied in water colour. The method is sensitive to $\simeq$ 0·05 mg of Pb per cubic metre and ceases to be precise at about five times the threshold toxic limit (0·2 mg per cubic metre). Of the common metal fumes, those of Cd and Zn also form coloured complexes. The stains of Cd and Zn can be removed by immersing the developed paper in 10% (v/v) acetic acid for 30 sec. and washing it with water acidified with acetic acid. A stain due to Sb can be removed together with those due to Cd and Zn by using a 5% aq. soln. of Na H tartrate.

A. O. JONES

3928. **Rapid method of determining lead and zinc in bronze by potentiometric titration involving precipitation of the sulphides.** V. A. Kremer, E. I. Vaĭl', F. P. Frizyuk and L. S. Sonchik (Mining Inst., Inst. of Chem., Kharkhov State Univ.). *Zavod. Lab.*, 1958, **24** (12), 1440–1441.—The method is based on titration in alkaline soln. with Na_2S soln. with the apparatus previously described (*cf.* Kremer and Vaĭl', *Anal. Abstr.*, 1957, **4**, 2884). The sample (0·5 g) containing a few per cent. of Zn and Pb is dissolved in 3·8 ml of conc. HNO_3, and Cu is removed by treatment with Na acetate, hydrazine sulphate and NaOH soln. so that the soln. (50 to 60 ml) after filtration is $\simeq$ 2 *M* in NaOH and contains the Pb and Zn, together with Sn and Sb, but < 0·1% of Cu. If this amount of Cu cannot be neglected the soln. at 60° is first titrated at 400 to 450 mV with the Na_2S soln. Otherwise a few drops of the titrant are added to the soln. at 60°, an electrode (Ag_2S) that is kept exclusively for titrations of Pb is introduced, and titration is carried out rapidly to a potential of 600 mV and then slowly to 650 mV. The soln. is then treated with dil. H_2SO_4 soln. (*e.g.*, 10 ml of a soln. containing 0·44 g of H_2SO_4 per ml) to lower the NaOH concn. to 0·5 *M*, the temp. is raised to between 70° and 80°, Na_2S soln. (0·5 to 1·0 ml) is added, another electrode is introduced, and the soln. is titrated to a potential of 700 mV and then more slowly to 750 to 770 mV. The contents of Pb and Zn are found by the use of a calibration curve. The method takes only 20 30 min. and results are accurate to within ± 2 of the contents.

G. S. SMITH

3929. **Volumetric determination of chloride titanium and zirconium by a polarisation end-poi procedure.** D. Price and F. R. Coe (Res. Dep I.C.I. Ltd., Metals Div., Kynoch Works, Witto Birmingham, England). *Analyst*, 1959, **84**, 5 62.—The development of an amperometric pr cedure is described, an electrode pair of silve silver chloride being incorporated in a dead-st circuit. The sample (1 g) is dissolved with warmi in 60 ml of dil. H_2SO_4 (1 + 4) and 1 ml of fluor boric acid, and the soln. is oxidised by addition a slight excess of HNO_3 and raising it to b.p. T electrodes are immersed in the cooled soln., t circuit is completed and the liquid is titrated wi 0·01 *N* $AgNO_3$. Near the end-point, galvanomet readings are taken after each addition of 0·1 ml $AgNO_3$ soln. The graph reverses its direction the end-point. A reagent blank is necessar expecially when the Cl^- content of the sample < 0·01%. Soln. containing 1 mg of Cl^- can b titrated to an accuracy within ± 1% and as litt as 0·5 μg of Cl^- can be determined with a precisio better than 20%. Preliminary work suggests tha the procedure is applicable to the determination Ag in certain alloys.

A. O. JONES

3930. **Analytical aspects of some organic acid II. Gravimetric estimation of zirconium by *m* phenylenedioxydiacetic acid.** C. S. Pande an T. S. Srivastava (Chem. Dept., Lucknow Univ India). *Z. anal. Chem.*, 1959, **165** (5), 343–34 (in English).—*Procedure*—To the boiling zirconiu soln. are added NH_4NO_3 (5 g per 100 ml of soln and a hot soln. of 2% *m*-phenylenedioxydiacet acid (**I**) in HNO_3 medium, with constant stirrin The concn. of HNO_3 in this final mixture adjusted to 0·08 *N* and the soln. is slowly heate for a few minutes and set aside on a water bat The flesh-coloured ppt. is allowed to settle an collected on Whatman No. 42 filter-paper, the washed with 0·1% **I** soln., dried and ignited the oxide. Many metal ions, *e.g.*, Cu^{2+}, Be^{2+}, Mg^{2+}, Ca^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+}, UO_2^{2+} and Mn^{2+} are not pptd. with **I**, but Al^{3+}, Ce^{3+}, Cr^{3+} a Fe^{3+} interfere to some extent and are remov by double pptn.; Sn^{2+}, Sn^{4+}, $V_2O_2^{4+}$ and Th interfere. Results for the determination of Zr the presence of foreign ions are tabulated.

B. B. BAUMINGER

3931. **Analytical chemistry of zirconium. Enrichment of zirconium as negatively charg sulphate complex on the strongly basic ion exchang Amberlite IRA-400 and its separation from thoriu titanium, iron, aluminium and many other elemen** J. Korkisch and A. Farag (II Chem. Inst., Un Wien, Austria). *Z. anal. Chem.*, 1959, **166** (81–88.—In 0·1 *N* H_2SO_4 Zr forms a negativ charged complex which is very firmly adsorbed Amberlite IRA-400; after elution with 4 *N* H Zr is titrated with EDTA. Excess of SO [< 10 g of Na_2SO_4 or $(NH_4)_2SO_4$ per mg of does not interfere, but Cl^- and NO_3^- must exceed 250 mg and 100 mg, respectively, per of Zr; PO_4^{3-} interfere. The method permits separation of Zr (1 mg) from Mg, Ca, Al, Cu, Cd, Ti, Cr, Mn, Fe^{3+}, Co, Ni and rare-earth me (100 mg of each) and Th (10 mg), but Sn^{2+} (1 V^{5+} (10), Mo^{6+} (10), W^{6+} (0·5) and UO_2^{2+} (0·5 are also adsorbed, and Mo and W interfere in

DTA titration. *Procedure*—Pass the soln. of $ZrOCl_2$ in 0·1 *N* H_2SO_4 (100 ml containing 1 mg Zr and 1 g of Na_2SO_4) down a column of mberlite IRA-400 (sulphate form free from Cl^-) 0·5 ml per min. Wash with 0·1 *N* H_2SO_4 (50 ml) id elute with 4 *N* HCl (100 ml). Evaporate the uate, carefully remove H_2SO_4 by heating latinum crucible), and ignite the residue till free om organic matter. Dissolve the ZrO_2 in HF few ml) and evaporate to dryness; dissolve the ZrF_4 in *N* HCl (10 ml), add a 0·1% soln. of $SnCl_2$ *N* HCl (1 drop), warm briefly, and add 0·1% olochrome violet R soln. (0·5 ml). Titrate the ot orange soln. with 0·001 *M* EDTA in the presence $ThCl_4$ soln. (a few ml containing 25 mg of Th er ml in *N* HCl).

III. A new method for the concentration of rconium with strongly basic anion exchangers id its application to the determination of zirconium silicate rocks. J. Korkisch and A. Farag. *id.*, 1959, **166** (3), 170–180.—Zirconium is deter-ined in the presence of PO_4^{3-} by adsorption on mberlite IRA-400 or Dowex 1 or 2 anion-exchange sin (sulphate form) from a soln. 0·1 *N* in H_2SO_4 id 0·5% in NaF, and subsequent elution with HCl *N*). Calcium, Ba, Sr and rare-earth metals are eviously separated as double fluorides by addition $AlCl_3$ soln., and Ti is eluted with H_2SO_4 (0·1 *N*) ntaining H_2O_2. Procedures are described for the termination of Zr in the presence of Th, Mo, W d V. Samples of silicate rock are fused with Na_2CO_3, extracted with water, and filtered, and e residue is treated with HF before proceeding th the separation. The final determination is ade volumetrically with EDTA, with Solochrome olet R as indicator.

IV. The adsorption of zirconium - ascorbic acid mplexes on strongly basic anion exchangers. Korkisch and A. Farag. *Ibid.*, 1959, **166** (3), 1–185.—At pH ≃ 4, Zr may be retained by a ongly basic ion-exchange resin (ascorbate form) the ascorbic acid complex. The presence of g of NaCl, Na_2SO_4 or $NaNO_3$ in 100 ml of soln. uses incomplete recovery. The influence of pH the recovery is discussed. J. P. Stern T. R. Andrew

3932. **Precision studies on the determination of rogen in zirconium and Zircaloy.** E. L. Shirley, R. Ciaranello and G. R. Smith (Knolls Atomic wer Lab., Schenectady, N.Y.). *U.S. Atomic ergy Comm.*, Rep. KAPL–M–ELS–10, 1958, pp.—In an attempt to measure and possibly prove the accuracy and precision of the routine termination of N in zirconium and zirconium ys, a study was made of NH_3 analyses using h micro- and macro-distillation apparatus. covery data are shown for variations in the mical procedure as they effect the accuracy and cision of the determination.

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3933. **Use of a cathode discharge for the ermination of impurities in zirconium oxide.** Yu. I. Korovin and L. V. Lipis. *Optika i ktroskopiya*, 1958, **5** (3), 334–337; *Ref. Zhur., m.*, 1959, (5), Abstr. No. 15,102.—The usual ngement of cathode and circulating He, which urified by passage through a carbon trap and ed by liquid N, is employed. The pumping em consists of a series of connected pumps. e graphite cathodes lead into the quartz harge tube which is provided with a water er. The discharge tube is supplied from a high-voltage rectifier with an output of 1000 V. The optimum wt. of the sample is 30 to 50 mg. The sensitivity of the determination is related logarithmically to the amount of sample. With a current of 200 to 1400 milli-amp., only a few lines of Zr accompany the lines due to impurities. The sensitivity of the determination for V, K and Ti is 1×10^{-3} %, for Al, Fe, Co, Si, As, Na, Ni, Sn, Pb, Sb, Cr and Zr it fluctuates between 1×10^{-4} % and 3×10^{-4} %, but for Ag, Cu, Mn, Mg, Cd and Be it is 1×10^{-5} %. Because of the stability of the discharge, quant. determination of most of the elements mentioned is possible without using internal standard lines. The spectra of 25 samples can be taken in one day. K. R. C.

3934. **Gravimetric and photometric determination of thorium in uraninites by means of anthranilic acid.** Yu. V. Morachevskiĭ and I. A. Tserkovnitskaya (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), 55–59.—Thorium can be quant. pptd. in the presence of U, Pb and rare-earth elements from nitrate soln. of pH 4·2 to 5·6 by anthranilic acid (**I**). To determine Th in uraninite, dissolve the sample (25 to 400 mg) in HNO_3 (1:5) (10 ml), heat to remove oxides of N, add water (20 ml) and hydroxyammonium chloride (1 to 2 g) (to hold U in solution), and add ammonium acetate (0·5 g) and 1% aq. NH_3 to pH ≃ 4·5 (methyl orange). Heat to boiling and precipitate Th with a 0·35% aq. soln. of **I** (20 ml). Set aside for 15 min., filter off and wash the ppt. with hot water, and re-precipitate by dissolving it in 25 ml of HCl (1:10), adding hydroxyammonium chloride (1 g), adjusting the pH to ≃ 4·5 as indicated above, and adding the soln. of **I**. The ppt. can be ignited and weighed as ThO_2, or Th determined photometrically as follows. Wash the ppt. (≡ 0·2 to 0·5 mg of ThO_2) with hot water and ethanol, dissolve it in 0·1 *N* HCl (25 ml), and make up to 50 ml. To a 10- or 20-ml aliquot add 4% $NaNO_2$ soln. (1 ml) and a 1% soln. of 2-naphthol in 5% NaOH soln. (1 ml), make up to 50 ml and measure the extinction with a blue filter. The content of ThO_2 is found from a calibration curve. The error is 3 to 5%. C. D. Kopkin

3935. **Determination of microgram quantities of thorium in Zircaloy II by X-ray fluorescence spectroscopy with ion-exchange membranes.** W. S. Horton and W. D. Moak (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL–M–WSH–4, 1959, 11 pp.—The sample is dissolved in a mixed acid (HBF_4 - H_2SO_4 - HCl). An ion-exchange membrane, after being stirred in the soln., is placed in the X-ray fluorescence spectrometer, and the main Th emission line is excited by a primary beam of X-rays from a tungsten target tube.

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3936. **Ion-exchange method for the separation of thorium from rare earths and its application to monazite analysis.** R. A. Nagle and T. K. S. Murthy (Chem. Div., Atomic Energy Estab., Trombay, Bombay, India). *Analyst*, 1959, **84**, 37–41.—Cerium was chosen to represent the rare-earth elements of its group, from which Th can be separated by adsorption on a strongly basic anion-exchange resin. The soln. containing Th and Ce^{III} as sulphates is diluted to 75 ml and the pH is adjusted to ≃ 2·3. The ion-exchange column (15 mm int. diam. × 10 cm) is of Amberlite

IRA-100 (sulphate form). The soln. is passed through at 2·5 ml per min. and the column is washed with acidified water at 5 ml per min. In the combined eluate and washings Ce is determined colorimetrically or volumetrically. The Th is eluted with 2 *N* HCl and pptd. from the eluate with aq. NH_3. The collected ppt. is dissolved in the minimum amount of dil. HCl and Th is determined by pptn. as oxalate; Fe^{3+} tend to be adsorbed and eluted with the Th, but most are removed during the oxalate pptn. Adsorption may be prevented by reduction to Fe^{2+} with SO_2. Up to 50 mg of U as U_3O_8 can be tolerated as it is removed during the oxalate pptn., and, under the conditions described, 50 mg each of NO_3^- and Cl^- and 1 g of SO_4^{2-} as sodium salts do not interfere. To apply the method to monazite, a procedure is described for the preliminary removal of PO_4^{3-} by fusion with KHF_2 and conversion of the rare earths into sulphates. A. O. Jones

3937. **Separation of thorium, zirconium and niobium as sulphates by anion exchange.** F. Molnár and G. Tóth (Dept. of Chem., Central Res. Inst. for Physics, Hungarian Acad. Sci., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **19** (1), 75–81 (in English).—Distribution coeff. for ^{234}Th, ^{95}Zr and ^{95}Nb soln. in contact with Mykion PA (a strongly basic resin of quaternary amine type) were determined. On passing a mixture of the metal ions through a column (7·6 cm × 0·8 cm) of resin, in a soln. 0·1 *M* in Na_2SO_4 and 0·03 *M* in H_2SO_4, and eluting with the same solvent, Th was quant. removed; the flow rate was 0·22 ml per sq. cm per min. Zirconium was separated from Nb either by eluting with *M* HCl, followed by 4 *M* HCl to remove Nb, or by elution with 0·2 *M* H_2SO_4 from a longer column (18 cm × 0·8 cm) at 0·1 ml per sq. cm per min., when successive removal of Zr and Nb took place.
P. D. Parr-Richard

3938. **Polarographic determination of nitrate and nitrite by induced reaction.** Hiroshi Hamaguchi, Jinzo Hashimoto and Norio Fukushi (Fac. of Sci., Tokyo Univ. of Educ., Koishikawa). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1958, **79** (12), 1482–1485.—In 0·1 *M* $NaClO_4$ soln. containing Cr^{III} - α- (or β-) alanine, or Cu - glycine, NO_3^- and NO_2^- give a distinctive induced wave of irreversible nature at − 2·0 V *vs.* the S.C.E. (their $E_{\frac{1}{2}}$ values are identical). The wave height is proportional to the concn. of NO_3^- and NO_2^- for < 0·0010 and < 0·0014 *M*, respectively, being almost independent of the pH (2 to 10). A mixture of 50 ml each of 0·1 *M* $NaClO_4$, 0·1 *M* $Cr(ClO_4)_3$ and 0·3 *M* β-alanine is heated on a water bath for 2 hr., then cooled and made up to 500 ml with 0·5 *M* NaOH. A 2-ml aliquot is mixed with 0·1 *M* $NaClO_4$ (10 ml) and the sample soln. (12 ml) and the polarogram is recorded. No interference results from SO_4^{2-}, PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^-$, MnO_4^-, CrO_4^{2-}, AsO_3^{3-}, CO_3^{2-}, halide ions, alkali-metal ions, NH_4^+, alkaline-earth ions or dissolved oxygen. K. Saito

3939. **The absorption of arsine by potassium iodate and its utilisation for the polarographic determination of arsenic.** D. Pavlov and N. Elenkova. *Izv. Khim. Inst. Bulg. Akad. Nauk*, 1958, **6**, 33–43.—The arsenic is converted to AsH_3 which is absorbed in a soln. of composition 2 *N* H_2SO_4, 0·0075 *N* KIO_3 and 0·0037 *M* $Ce(SO_4)_2$ to which tartaric acid is added immediately before the experiment to make the soln. 0·011 *M*. The resulting soln. is polarographed. N. E.

3940. **Determination of bismuth, lead and cadmium in vanadium and niobium.** V. A. Nazarenko and E. A. Biryuk (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (1), 28–30.—*Procedure with vanadium*—The sample (1 g) is dissolved in 7 ml of conc. HNO_3 and the soln. is evaporated to fuming with 5 ml of conc. H_2SO_4, then diluted with water and again evaporated. The cooled and diluted soln. (50 ml) is neutralised to Congo red with 20% NaOH soln., and 3 g of ammonium hydrogen tartrate is added followed by sufficient NaOH soln. to give a red colour with alizarin yellow R[G] paper. The cooled soln. is mixed with 2 ml of 2% KCN soln. and 5 ml of 1% Na diethyldithiocarbamate soln. and extracted with $CHCl_3$ (2 × 5 ml), which extracts Bi, Cd, Pb and Tl^{3+}. The $CHCl_3$ soln. is washed with 5 ml of 5% KOH soln. and then shaken with 0·2 *N* HCl (2 × 2·5 ml), which extracts the Pb and Cd. To determine Bi, the $CHCl_3$ soln. is evaporated to dryness, the residue is treated with 0·5 ml of 2 *N* H_2SO_4 and 0·5 ml of 30% H_2O_2 and evaporated just to fuming. After addition of 3 ml of water, 0·5 ml of 5% ascorbic acid soln., 0·3 ml of 15% KI soln. and 0·4 ml of *iso*butyl methyl ketone, the mixture is shaken and the colour of the ketone layer is compared with that of standards containing from 0 to 5 μg of Bi. To determine Pb, one half of the aq. extract containing Pb and Cd is treated with 2·5 ml of 0·2 *N* HCl, 2 ml of 4·5 *N* aq. NH_3, 0·4 ml of 0·5% KCN soln. and 0·5 ml of 20% hydroxyammonium chloride soln., and then shaken with 1 ml of a 0·002% soln. of dithizone in $CHCl_3$. The colour of the extract is compared with that of standards containing 0 to 3 μg of Pb. To determine Cd, the other half of the soln. is mixed with 1·5 ml of 10% NaOH soln., 0·1 ml of 0·5% KCN soln. and 1 ml of 0·002% dithizone soln. The colour of the extract is compared with that of standards containing 0 to 3 μg of Cd. *Procedure with niobium*—The sample (1 g) is treated with 5 ml of HF soln. and then dropwise with 3 ml of conc. HNO_3, and the soln. is evaporated until the appearance of a ppt., which is then dissolved by the addition of 1 to 2 ml of HF soln. The soln. is poured into a platinum dish containing 7 g of ammonium hydrogen tartrate dissolved in 30 ml of 20% KOH soln. A further amount of KOH soln. is added to give a red colour with alizarin yellow R[G] paper and the soln. is then treated as described for vanadium for the determination of Pb and Cd. To determine Bi, the sample of niobium (1 g) is heated in a Kjeldahl flask with 4 g of K_2SO_4 and 10 ml of conc. H_2SO_4 until the metal is dissolved and S is removed from the walls. The cooled mass is mixed with a soln. containing 7 g of citric acid and 1·5 ml of 30% H_2O_2 and the flask is washed once with water. The cooled soln. is treated with 1·5 g of $K_2S_2O_5$ added in small portions and then with 22 ml of 12 *N* aq. NH_3 and diluted with water to 100 ml. The soln. is extracted with a soln. of diethyldithiocarbamic acid in $CHCl_3$ (3 × 10 ml), prepared as described previously (*cf.* Nazarenko *et al.*, *Anal. Abst.*, 1958, **5**, 2954), and the extracts are washed with 0·5 *N* H_2SO_4 (3 × 10 ml). After re-extraction with 6 *N* HCl (3 × 5 ml) the resulting soln. is evaporated to dryness and the residue is treated with 0·5 ml of 2 *N* H_2SO_4 and 0·5 ml of 30% H_2

d heated just to fuming. The residue is treated th 1·5 ml of water and 1 ml of 5% oxalic acid n. Standards containing 0 to 5 μg of Bi together with 0·5 ml of 2 *N* H_2SO_4 and 1 ml of 5% alic acid soln. in 2·5 ml are prepared simulneously. Each soln. is mixed with 0·5 ml of % ascorbic acid soln., 0·1 ml of 9% thiourea n., 0·3 ml of 15% KI soln. and 0·4 ml of *iso*tyl methyl ketone. The colours of the ketone ers are compared within 5 min. of the mixing.

G. S. Smith

3941. **Photometric 1:10-phenanthroline method determining vanadium in metallic chromium.** Ya. Yakovlev and G. P. Razumova (Central i. Res. Inst. of Ferrous Metallurgy). *Zavod. b.*, 1958, **24** (12), 1430–1431.—Small amounts of (0·0016 to 0·0080%) in chromium metal are termined by the following method. The sample g) is dissolved in 40 ml of dil. H_2SO_4 (1:1) with bsequent addition of HNO_3. The soln. is evaporad to fuming and diluted to between 200 and 0 ml. The cooled soln. is mixed with 1 ml of 1% Cl_3 soln. and sufficient 1% $KMnO_4$ soln. to give pink colour, and then treated with cupferron to ecipitate Fe and V. The ppt. is collected and shed with cold 10% H_2SO_4 soln. containing 1·5 g cupferron per litre, then ashed and fused with $_2CO_3$. The filtered soln. of the melt is acidified th HCl, then evaporated to between 70 and ml, cooled and diluted to 100 ml. An aliquot ml) is treated with 2 ml of conc. HCl and 5 ml a freshly prepared 4% soln. of $NaNO_2$ and set de for 10 to 15 min. The excess of $NaNO_2$ is troyed during 20 to 25 min. by addition of 2 ml a saturated soln. of urea; 1 ml of a soln. of Cl_3, containing $\simeq$ 0·1 mg of Fe, and 1 ml of % 1:10-phenanthroline soln. are added and the n. is made just alkaline to Congo red paper with NH_3 soln. After 25 to 30 min. the soln. is ated to 100 ml with a 2% soln. of borax and extinction is measured with a green filter.

G. S. Smith

942. **Determination of niobium in uranium-bium alloys.** T. C. Bryson, G. W. Goward and D. Hartmann (Westinghouse Electric Corp., tis Plant, Pittsburgh). *U.S. Atomic Energy m.*, Rep. WAPD-CTA-GLA-146, April 1956, 11. Jan. 1959, 4 pp.—Niobium is determined as peroxy complex in conc. H_2SO_4 by photometric surement at 360 mμ. Uranium interference is celled by using a soln. of the sample for reference the photometric measurement. The method ers the determination of Nb in uranium-bium alloys made from pure melting stock. ybdenum interferes and must be removed. conium does not interfere. The calibration e covers a concn. range from 0·2 to 5·0 mg.

Nucl. Sci. Abstr.

943. **Determination of niobium in the presence ungsten by using cupferron.** A. I. Ponomarev A. Ya. Sheskol'skaya (A. A. Baĭkov Inst. of allurgy, Acad. of Sci., USSR, Moscow). *Zhur. l. Khim.*, 1959, **14** (1), 67–70.—The method is d on pptn. of Nb with cupferron (**I**) from a dly acid medium containing oxalic acid (**II**), monium oxalate (**III**) or tartaric acid (**IV**). To rmine Nb in the alloy W - Si - Nb, add HF 3 ml) to the sample (0·1 g) in a platinum dish, fully add HNO_3 to complete the dissolution, orate to dryness on a sand bath and fuse the ue with $K_2S_2O_7$ (2 to 3 g). To the cold melt add a 4% soln. of **II**, **III** or **IV** (30 to 40 ml), heat on the sand bath, add water (30 to 40 ml), and continue heating to complete dissolution. Make up the soln. to 150 to 200 ml, add 5 drops of conc. HCl, and precipitate Nb with 20 to 25 ml of a 3% aq. soln. of **I**, added slowly and with stirring. Add a little macerated filter-paper, filter, and wash the ppt. (× 6 or 8) with cold water containing 20 ml of 3% **I** soln. and 1 ml of conc. HCl per litre. Ash and ignite the ppt. at 500° to 600°, fuse the residue with $K_2S_2O_7$ (1 to 2 g), add to the cold melt a 4% soln. of **II** (20 to 30 ml), and re-precipitate as described above. Ash, ignite the residue at 1000°, cool in a desiccator and weigh as Nb_2O_5. The method was tested with the radioactive isotope ^{185}W. The content of W in the Nb_2O_5 increases with the original proportion of Nb; with 30% or more of Nb in the alloy some W passes through into the Nb_2O_5. To determine Nb in tungsten steel or other materials containing Fe, Ti, V and other elements pptd. by **I**, first precipitate Nb and W with tannin in the presence of ascorbic acid (*cf. Anal. Abstr.*, 1958, **5**, 466), and then separate them as described above.

C. D. Kopkin

3944. **Determination of niobium and tantalum in titanium-base alloys.** J. L. Hague and L. A. Machlan (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand.*, 1959, **62** (2), 53–57.—The sample (5 g) is dissolved in a mixture of HCl and HF at 40° to 50° and the soln., after oxidation with HNO_3 (1 to 2 ml), is passed slowly through a column (12 in. × 1 in.) of Dowex-1 resin ($\simeq$ 200 mesh). The interfering Ti, Fe, V and Sn are removed by elution with a soln. of NH_4Cl - HCl - HF and the eluate is discarded. Niobium is then collected by elution with an NH_4Cl - HF soln., and finally Ta by elution with a slightly acid soln. of NH_4Cl and NH_4F. The F^- in the two eluates are then complexed by the addition of H_3BO_3 and, after adding 70 to 85 ml of HCl and 80 ml of H_2O, niobic and tantalic acids are pptd. with cupferron, and washed and ignited to Nb_2O_5 and Ta_2O_5, respectively. The accuracy is high, but the procedure requires 2 to 3 days. Impurities in the final ppt. are usually < 0·2 to 0·3 mg.

W. J. Baker

3945. **Analysis of tantalum - niobium binary alloys by reflection of β-radiation.** V. B. Gaĭdadȳmov and L. I. Il'ina. *Trudȳ Komiss. Anal. Khim.*, 1958, **9** (12), 240–248; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 27,117.—The method developed is based on the difference in intensity of the β-radiations from Ta and Nb, and a comparison of the intensity of the reflected β-radiations from test alloys and standards. The radioactive isotope must be very carefully selected as it exerts a considerable influence on the accuracy of the determination. The determination of the reflection coeff. of different metals shows that, with an increase in the energy max. of the incident β-radiation, the intensity of reflected β-radiation is considerably increased; for energy < 1 MeV there is direct proportionality. The best radio-isotopes both as regards a high energy max. of β-radiation, long half-life and purity of radiation are ^{204}Tl and ^{90}Sr. The intensity of β-radiation reflected from Nb and Ta increases with an increase in the thickness of the aluminium filter to 130 mg per sq. cm when the radioactive isotope ^{204}Tl is used. The alloy is powdered, heated at 800° to oxidise Nb and Ta to their pentoxides, and a sample ($\simeq$ 1 g) is compressed in a Plexiglas cell at 100 kg per sq.

cm. The compressed samples are analysed by comparison with artificially prepared standards of Nb_2O_5 and Ta_2O_5. To attain a high degree of accuracy, a differential ionisation chamber with a constant-current amplifier working at saturation point is used instead of a Geiger - Müller counter. The activity of ^{204}Tl is 30 mC. The minimum thickness of the test material necessary for the production of reproducible results is 100 mg per sq. cm. This method can be used to determine up to 0·5% of Ta in an alloy; the error of determination is < 0·5% (absolute). K. R. C.

3946. **Kinetic methods of quantitative analysis. V. Quantitative determination of tantalum.** K. B. Yatsimirskiĭ, O. M. Drobÿsheva and V. I. Rigin (Ivanovo Chem.-Technol. Inst.). *Zhur. Anal. Khim.*, 1959, **14** (1), 60–62.—The catalysis of the oxidation of KI by H_2O_2 is used to determine Ta. Soln. of 0·01 *M* KI, 0·01 *M* H_2O_2, 0·2% starch and *M* HCl (free from heavy metals) are used. The Ta soln. is prepared by fusion of Ta_2O_5 with $K_2S_2O_7$ and extraction with satd. ammonium oxalate soln., and the concn. determined by the tannin method. Mix in a 50-ml flask the soln. of Ta, KI, starch, HCl and water to a vol. of 40 to 43 ml, add the H_2O_2, make up to vol., mix and measure the extinction at regular intervals, zero time being the time of addition of the H_2O_2. The concn. are Ta 2×10^{-6} to 5×10^{-5} *M*, KI and H_2O_2 2×10^{-4} to 2×10^{-3} *M*, and HCl 0·1 to 0·5 *M*. The concn. of Ta is found from the equation—

$$d[I_2]/dt = (x_0 + x_1 C_{H^+}).C_{I^-}.C_{H_2O_2}.C_{Ta},$$

where C_{I^-}, $C_{H_2O_2}$, C_{H^+} and C_{Ta} are the concn. of the respective substances, and x is the rate constant for the reaction. No interference is caused by the usual cations and anions (K^+, Na^+, Mg^{2+}, Ca^{2+}, Al^{3+}, Zn^{2+}, Cl^-, SO_4^{2-}, ClO_4^-) or Ti, Zr or Nb. The presence of F^- slows down the reaction, while NO_3^- accelerates it owing to the catalytic effect of traces of nitrite.

C. D. Kopkin

3947. **Spectrographic determination of bismuth, cadmium, tin, lead and antimony impurities in tantalum.** E. I. Zakharov, L. V. Lipis and K. I. Petrov. *Zhur. Anal. Khim.*, 1959, **14** (1), 135–136.—The Ta is oxidised by heating in a muffle-furnace at 600° to 700° for 1·5 to 2 hr. and the impurities are determined spectrographically at 1400° by the evaporation method from graphite crucibles, oxides of the impurities being completely volatile while Ta_2O_5 is practically non-volatile. The distilled oxides are condensed on to copper electrodes. The internal standard is 0·01% of Tl. The experimental conditions are—a condensed spark discharge, a 2-mm spark gap, and 10 sec. exposure. The pairs of lines used are—Bi 3067·72 and Tl 2918·32; Sn 2839·99 and Tl 2918·32; Pb 2833·07 and Tl 2918·32; Cd 2833·02 and Tl 2580·14; and Sb 2598·53 and Tl 2580·14 A. The sensitivities are—Bi and Cd 10^{-5} %, Pb and Sn 3×10^{-5} %, and Sb 10^{-4} %. The coeff. of variation of the determinations are—Bi ± 8%, Pb and Sb ± 10%, and Cd and Sn ± 11%. C. D. Kopkin

3948. **Detection of traces of sulphur.** W. Noddack and I. Noddack (Staat. Forschungsinst. f. Geochem., Bamberg). *J. prakt. Chem.*, 1957, **5**, 274–281.—Traces of $BaSO_4$ too small to filter and weigh can be detected by the procedure described. The $BaSO_4$ is either co-pptd. with a carrier or pptd. in a centrifuge tube and reduced to BaS by heating in a stream of H. The BaS is decomposed with HCl and the H_2S produced is carried in a stream of N through a filter-paper impregnated with Ag or Pb acetate. By measuring the intensity of the stain, 10^{-4} mg of S can be determined to within 5%. Suitable apparatus is described. With a reflection photometer the limit of detection is 5×10^{-7} mg of S. If, instead of acetate, the paper is coated with the emulsion removed by hot water from positive film, the stain can be developed and fixed by the usual photographic methods, and the sensitivity is increased by a factor of 500.

G. Burger

3949. **Determination of microgram quantities of sulphur in technical gases.** E. Abel and H. Barth (Rheinisch-Westfälischen Hochsch., Aachen). *Arch. Eisenhüttenw.*, 1958, **29** (11), 683–684.—The suitability of Stratmann's micro-analytical method for the determination of SO_2 in atmospheric air (*cf. Anal. Abstr.*, 1955, **2**, 1056) was investigated for application to determinations of total S in technical gases, and the following modifications were made. Electrical heating for the adsorption tube and the platinum contact and U-shaped contact tubes were used. Methylene blue indicator is recommended, since it is not affected by the various carrier gases which are evolved during the process. The method can be conveniently used for solids and gaseous substances. The modified apparatus is illustrated. L. M. Riegelhaupt

3950. **Density - composition tables for aqueous solutions of sulphuric acid.** British Standards Institution (2 Park St., London, W.1). B.S. 753: 1959, 67 pp.—This is a revision of the density - composition tables published as B.S. 753:1937. The text has been revised to accord with the latest hydrometer standard (B.S. 718:1953) but the tables have not been re-calculated as readings can readily be corrected by methods described.

3951. **Use of 4-amino-4′-chlorodiphenyl as reagent for the determination of sulphate.** R. Belcher and W. I. Stephen (Birmingham Univ., England). *Anal. Chim. Acta*, 1959, **20** (2), 197–199.—Bengtsson's criticism (*cf. Anal. Abstr.*, 1959, **6**, 134) is answered and 4-amino-4′-chlorodiphenyl is claimed to be, in practice, the most sensitive known precipitant for SO_4^{2-}. H. N. S.

3952. **Titrimetric micro-determination of sulphate using lead nitrate as titrant and dithizone as indicator.** D. C. White (Res. and Dev. Dept., Distillers Co. Ltd., Epsom, Surrey, England). *Mikrochim. Acta*, 1959, (2), 254–269 (in English).—The method of Archer (*Analyst*, 1957, **82**, 208) has been adapted to the micro and sub-micro scale, and 0·16 to 1·6 mg or 4 to 30 μg of S can thus be determined. Halogens and phosphate interfere; the former are removed by evaporation with HNO_3 and the latter by pptn. as the magnesium ammonium salt. Nitrate interferes when present in large (5-fold) excess. Metals that form coloured complexes with dithizone (*e.g.*, Cu^{II}, Ag, Hg^{II}, Co^{II}, Ni^{II} and Fe^{III}) interfere and are removed by passage of the soln. through a column (80 mm long and 7 mm internal diam.) of Amberlite IR-120 (acid form). It is recommended to pre-treat the column three times with 2 *N* NaOH followed by H_2O and then 2 *N* HNO_3. Eight organic compounds (including sulphanilic acid and phenoxymethylpenicillin) were successfully analysed. B. B. Bauminger

953. **Complexometric determination of sulphate.** Rumler, R. Herbolsheimer and G. Wolf (Staatl. t. für Hygiene u. Infektionskrankheiten, Saarcken, Germany). *Z. anal. Chem.*, 1959, **166** 23–24.—Barium sulphate dissolves readily in tri- or mono-ethanolamine soln. with an excess EDTA. The excess of EDTA can then be ated with $MgCl_2$ soln. G. BURGER

954. **Polarography of quadrivalent selenium and ırium.** I. E. Bŷkov, A. I. Zelynskaya and S. Gorshkova. *Trudŷ Inst. Metallurg. Ural'skiĭ Akad. Nauk SSSR*, 1958, (2), 275–279; *Ref. ır., Khim.*, 1959, (7), Abstr. No. 23,090.—The sibility of obtaining separate waves of Se^{4+} Te^{4+} on one polarogram was studied. Coulo- ric measurements confirmed the 6-electron racter of the process of reduction of Se and Te. following basal soln. (pH 9) was found to be most suitable—0·5 *M* NH_4Cl, 0·5 *M* aq. NH_3, *M* Na_2SO_3 and 0·002% gelatin; at this pH Se Te form single polarographic waves adequately ırated from one another. The molar concn. of should not exceed that of Se. It is possible to ırograph Te - Fe in tartrate soln. and Te - Pb Se - Zn in a soln. containing EDTA (disodium . In the determination of Te in the presence Cu, polarography is carried out in an alkaline nide soln. In the analysis of the industrial lucts of Se and Te, pptn. is carried out with I_2 followed by oxidation with $KClO_3$ in the ence of HCl. K. R. C.

955. **Malonate complexes of chromium and and their analytical uses.** A. I. Cherkesov, S. Galkina and A. S. Mel'nikova. *Trudŷ akhansk. Tekh. Inst. Rŷbn. Prom. i Khozyaist.*, , (5), 132–142; *Ref. Zhur., Khim.*, 1959, (5), ır. No. 15,045.—It is shown that malonic acid s a complex with Fe^{3+}, in which the ratio of onic acid to Fe is 3:1. In the reaction of onic acid with Cr^{3+}, a complex is formed on ng which has a similar composition to the Fe plex; the soln. is much more intensely coloured z. extinction at 574 mμ) than the original soln. *Procedure for Cr in steel*—Dissolve the ple (1 to 2 g) in 20 to 40 ml of HCl (sp. gr. and evaporate the soln. to 10 to 15 ml; if a s ppt. forms (*e.g.*, carbides of W) add HNO_3 gr. 1·37) dropwise until the soln. has turned w. Dilute with hot H_2O (10 to 15 ml), filter, make up to 50 to 100 ml with H_2O; neutralise liquot (5 to 10 ml) with satd. Na_2CO_3 soln. it becomes turbid, add 0·5 *M* malonic acid o 20 ml), boil for 3 to 5 min., cool, make up ml with H_2O and measure photometrically. K. R. C.

956. **Spectrophotometric and kinetic methods etermining molybdenum in alloys.** K. B. imirskiĭ and I. I. Alekseeva (Ivanovo Chem.- Inst.). *Zavod. Lab.*, 1958, **24** (12), 1427–—A spectrometric method of determining MoO_4^{2-} is based on the absorption at 220 to 240 mμ e alkaline soln. The Beer - Lambert law is ed, the molar extinction coeff. is ≃ 7000, the tivity is 7×10^{-7} g of Mo per ml, and most elements do not interfere. With steel and l alloys containing from 0·5 to 5% of Mo, the le (0·4 g) is dissolved in conc. HCl with subse- addition of HNO_3, the soln. is evaporated to ess, and the residue is dissolved in water or HCl; an aliquot (20 ml) of the filtered and ed soln. (200 ml) is heated with 5 to 7 ml of 20% NaOH soln., the filtered soln. is diluted to 200 ml and the extinction is determined at 220 to 240 mμ. The content is calculated by means of a calibration curve obtained by use of standards. The error is within ± 5% of the content. The kinetic method is based on the catalytic action of MoO_4^{2-} on the oxidation of I^- by H_2O_2 in acid soln. The interference of Fe and Cu is prevented by the use of EDTA. The sample of steel (0·3 g) containing > 1% of Mo is dissolved as described above and an aliquot of the filtered soln. in a 50-ml flask is treated with H_2SO_4 so that the final soln. will be 0·1 *N* in acid, and then with 5 ml of 0·05 *M* EDTA (disodium salt), KI soln. to give a concn. of 0·0002 *M*, starch soln., and sufficient H_2O_2 soln. to give a concn. of 0·001 *M* after dilution to 50 ml. The change of extinction is determined during 10 min., readings being taken every minute in a photo-electric colorimeter with a green filter. The results are plotted and the slope of the line is measured. For calibration, the soln. (50 ml) contains 2 to 3 drops of 10% $FeCl_3$ soln., 5 ml of 0·05 *M* EDTA (disodium salt), KI soln. (as above), starch, H_2SO_4 (as above), $(NH_4)_2MoO_4$ to give a final concn. of between 10^{-7} and 10^{-8} *M*, and H_2O_2 soln. (as above, added after suitable dilution). A calibration curve of slope *vs.* concn. is constructed. The method is sensitive to 10^{-8} g of Mo per ml. G. S. SMITH

3957. **Spectrographic analysis of tungsten metal powder.** R. Dyck and T. J. Veleker (Chem. and Metallurg. Div., Sylvania Electric Products, Inc., Towanda, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 390–392.—For the rapid determination of Al (0·0001 to 0·01%), Ca (0·0001 to 0·005%), Fe (0·0001 to 0·02%), Mg (0·0001 to 0·005%), K (0·003 to 0·1%) and Si (0·0002 to 0·1%) in tungsten metal, the metal powder is mixed with graphite and an internal standard (Li_2SO_4 for K, nickel powder for other elements). Graphite suppresses the tungsten spectrum initially, by formation of tungsten carbide, while the impurities to be determined show peak intensities during this initial period. Graphite electrodes and high-voltage a.c. arc excitation are used. P. D. PARR-RICHARD

3958. **Spectrographic determination of aluminium, iron, potassium and silicon in tungstic oxide.** T. J. Veleker and R. Dyck (Chem. and Metallurg. Div., Sylvania Electric Products, Inc., Towanda, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 387–390.—To determine Al (0·005 to 0·15%), Fe (0·008 to 0·1%) and Si (0·02 to 0·5%), W_4O_{11} is oxidised at 750° and mixed with graphite and anhyd. sodium tungstate; the addition of tungstate suppresses interference due to irregular formation of tungsten carbide. After pelleting, samples are sparked, with graphite electrodes; tungsten serves as an internal standard. For K (0·025 to 0·35%), W_4O_{11} is oxidised as before, mixed with Li_2SO_4 to provide an internal standard, and loaded into brass electrodes; high-voltage a.c. arc excitation is employed. P. D. PARR-RICHARD

3959. **Determination of tungsten and iron in tungsten-containing materials.** B. S. Khristoforov and Z. M. Arkhipova. *Vestn. Leningr. Univ.*, 1959, No. 4, *Ser. Fiz. i Khim.*, (1), 139–140.—Soln. of $FeCl_3$ containing 19·70 mg of Fe per ml and of Na_2WO_4 containing 7·83 mg of WO_3 per ml were mixed in different proportions, made up to 250 ml, and boiled with HCl for 5 to 10 min. to precipitate the bulk of the H_2WO_4. The pptn. of W was

completed by the addition of cinchonine. The tungsten ppt. were filtered off and the filtrates, after evaporation to 150 to 200 ml, were reduced with $SnCl_2$ soln. and the Fe content determined by the usual dichromate method. The results for several concentrates are tabulated, and the method is shown to be both rapid and reliable. K. R. C.

3960. **Determination of tungsten and niobium in alloys.** Yu. I. Bŷkovskaya. *Trudy Komiss. Anal. Khim.*, 1958, **9** (12), 323–328; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 27,115.—Methods for the determination of W and Nb in iron-, nickel- and cobalt-base alloys were tested by using ^{185}W and ^{59}Nb. Results obtained with $HClO_4$ (Schoeller *et al.*, *Analyst*, 1935, **60**, 506) and HCl (Golubtsova, *Zhur. Anal. Khim.*, 1951, **6**, 357) for the dissolution of the alloy and subsequent hydrolysis of W and Nb were compared, and the completeness of separation of W and Nb by using an $MgSO_4$ - NH_4Cl - aq. NH_3 mixture was determined. With $HClO_4$ or HCl, the hydrolysis of W and Nb was practically complete. With 1-g samples containing 0·21 to 0·36 mg of W and 0·047 to 0·11 mg of Nb, respectively, 0·4 to 0·7% of unhydrolysed W and 0·3 to 0·7% of unhydrolysed Nb, respectively, remained in soln. In the separation of W and Nb with the $MgSO_4$ mixture, W was only slightly occluded in the ppt. (0·2 to 0·5%, or 0·11 to 0·26 mg for a 1-g sample). Niobium was not pptd. quant.; 7 to 17% of the total content remained in soln. with W.
K. R. C.

3961. **Titrimetric determination of tungstate ions as lead tungstate.** H. Grubitsch, N. Özbil and K. Kluge (Inst. f. Anal.-Chem., Tech. Hochsch., Graz, Austria). *Z. anal. Chem.*, 1959, **166** (2), 114–120.—Optimum conditions are established for the precipitation - titration of WO_4^{2-} as $PbWO_4$, the end-point being indicated potentiometrically or preferably by adsorption indicators. Interference by NH_4Cl, NH_4NO_3, Na or K acetate, and complexing ions (tartrate, oxalate, citrate, PO_4^{3-}) is serious. With the specified mixed indicator, $>$ 30 mg of W is determined with an error $\gtrless \pm 1\%$ (mean error $\pm$ 0·1%). *Procedure*—Just acidify the WO_4^{2-} soln. with HNO_3 to bromothymol blue, boil to expel CO_2, and adjust with NaOH exactly to the green colour-change. Either titrate potentiometrically (tungsten - satd. calomel, or tungsten - platinum electrodes) at 50° with 0·05 *M* $Pb(NO_3)_2$; or preferably destroy the bromothymol blue with bromine vapour (and subsequent boiling), add the mixed azo-dye indicator ["Aminschwarzgrün B" (Agfa) and "Diaminechtscharlach 6 BS" (Cassella), 0·2% aq.], and titrate the soln. while it is cooling spontaneously. At the end-point, which is very sharp, the soln. is blue and the ppt. red.
J. P. Stern

3962. **Polarographic reduction of sexavalent uranium in sodium tripolyphosphate. II. Estimation of uranium.** P. R. Subbaraman, N. R. Joshi and J. Gupta (Nat. Chem. Lab., Poona, India). *Anal. Chim. Acta*, 1959, **20** (2), 190–194.—In the polarographic determination of UO_2^{2+}, interference from WO_4^{2-}, MoO_4^{2-}, Ti^{4+}, Cr^{3+}, Ni^{2+}, Zn^{2+}, Mn^{2+}, Co^{2+}, Pb^{2+} and Bi^{3+} is avoided by using an electrolyte that contains $Na_5P_3O_{10}$ (0·1 *M*) together with KNO_3 (*M*) and camphor (0·005%). To render the method even more generally applicable, preliminary pptn. of the UO_2^{2+} as ammonium uranyl phosphate (*cf.* Milner and Edwards, *Anal. Abstr.*, 1957, **4**, 2185) is carried out. The procedure is applied to tantaloniobate, after pptn. of th bulk of the Ta, Nb and Ti by boiling the slightl acid soln. of the sample. Monazite is decompose with conc. H_2SO_4, the mass is diluted and twic boiled with Na_2CO_3 soln. to extract the U. Th determination is completed as described above.
H. N. S.

3963. **The emission and absorption spectra uranium excited in a King furnace.** L. Bovey an H. Wise (A.E.R.E., Harwell, Berks., England A.E.R.E. Report C/R 2766, 1959, 199 pp.—A li containing 3554 emission and 2319 absorptio lines (together with arc, furnace and spark intens ties and assignment when known) is included. discussion on the application of the results to th term analysis of uranium and plutonium is given.

3964. **Resorcinol as an analytical reagent fo the spectrophotometric determination of uraniun** P. C. Jain and G. S. Rao (Univ. of Saugar, India *Anal. Chim. Acta*, 1959, **20** (2), 171–174.—Uran ions form an orange - red 1:2 complex wit resorcinol. The absorption max. is at 400 450 mμ and the max. colour develops at pH 4·7 The reaction can be used for the spectrophot metric determination of U, but several other ion interfere.
H. N. S.

3965. **Direct spectrophotometric determination microgram quantities of copper in high-purit uranium with oxalodihydrazide.** D. B. Stevančev (Inst. of Nucl. Sci. "Boris Kidrich", Belgrad Yugoslavia). *Z. anal. Chem.*, 1959, **165** (5), 348 354 (in English).—To evaluate the suitability oxalodihydrazide (**I**) for the determination of C a comparison has been made with dithizone (**I** and Na diethyldithiocarbamate (**III**). The reactio with **I** is as sensitive as with **II**, and is three tim as sensitive as with **III**. The time required f analysis is much less than with **II** and **III**, as t colour is measured in aq. medium and no extractio with organic solvents is necessary. *Procedure*— Two 1-g samples of U (containing 2 to 30 p.p. of Cu) are placed in two 50-ml beakers, dissolv in HNO_3 (1:1) and evaporated to dryness. Aft cooling, 4 ml of H_2O is added followed by 5 ml *M* ammonium citrate and the soln. are transferr to 25-ml flasks. The beakers are washed twi with 2-ml portions of H_2O, and 4 ml of conc. a NH_3 is added to each flask. The concn. of aq. N should be $\lessgtr 2$ *M*. The contents of one flask a made up to vol. and serves as a reference sol and to the other flask are added 2 ml of satd. a **I** soln. and 4 ml of acetaldehyde and the mixtu is made up to vol. The extinction is read after $\pm$ 5 min. at 542 mμ and referred to a calibrati curve. The interference of foreign ions, except Co, is negligible.
B. B. Baumingei

3966. **Polarographic determination of mangan [in steel and in vegetable matter] by anodic oxidat on a platinum electrode.** E. M. Skobets and N Belinskaya. *Nauch. Trudy Ukr. Sel'skokh. Ak* 1957, **9**, 467–472; *Ref. Zhur., Khim.*, 1959, Abstr. No. 23,057.—*Procedure for steel*—Dissc the sample ($\simeq$ 0·3 g) in H_2SO_4 (1:2), add cc HNO_3, boil for 1 to 2 min. and make up to 100 with H_2O; transfer the soln. (1 to 2 ml) to a pola graphic cell and polarograph in a basal soln 0·1 *M* HNO_3 (total vol. of soln. $\simeq$ 25 ml) wit platinum wire as the anode and a S.C.E. as cathode. The amount of Mn is calculated by standard addition method. The height of the w

rectilinear function of the Mn concn. whether soln. be acid or alkaline. *Procedure for vegetable ter*—Incinerate the dry substance (10 g) at 600° 800°, evaporate the ash with conc. HCl (1 to l) and evaporate the dry residue with HNO_3 l) (1 to 2 ml); repeat the HNO_3 treatment 2 to mes, add a mixture of *N* NH_4Cl and 0·2 *N* aq. $_3$ to the residue, filter and polarograph.

K. R. C.

967. **Cacotheline as a specific reagent for the ection of iron(II).** G. Gopala Rao, V. Narayana and G. Somidevamma (Andhra Univ., Waltair, ia). *Z. anal. Chem.*, 1959, **166** (1), 11–18 (in glish).—A soln. of cacotheline at pH 7·4 (0·1% phosphate - citric acid buffer) gives a specific ction with Fe^{2+}. A blue colour is produced ch rapidly fades in air. In a tube in the absence ir the limit of detection is 11 μg in 5 ml. On a t plate the limits of detection and dilution are μg and 1 in 1 × 10^5 and on paper 0·3 μg and 1·66 × 10^5. Ferric iron can be detected after uction with oxalate in sunlight. Other, more sitive, tests are reviewed. None is both specific free from interferences. G. Burger

968. **Analytical applications of complexones. . Spectrophotometric micro-determination of with 1:2-di-(2-aminoethoxy)ethane-NN′-tetra-ic acid.** F. Bermejo Martínez and M. Paz Castro). de Quím. Anal., Fac. de Cien., Santiago de mpostela, Spain). *Inf. Quim. Anal.*, 1959, **13** 1–7.—Traces of Fe^{3+} (up to 100 μg per ml) t with this reagent to give a stable yellow plex with the absorption of which at pH 4 to 5 vs Beer's law. Large amounts of phosphates, $^{2+}$, Ni^{2+}, Mn^{3+}, Co^{3+}, F^- and CN^- interfere, but $^{3-}$, acetates, tartrates, phthalates, NO_3^-, Cl^- Br^- do not. This method enables 0·25 μg of $^-$ per ml to be detected. G. H. Foxley

969. **Gravimetric and radiometric volumetric nods for the determination of iron with monium benzene- and naphthalene-seleninates.** . Alimarin and V. S. Sotnikov. *Trudy Komiss. Z. Khim.*, 1958, **9** (12), 213–218; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 27,063.—A gravi-ric method was developed for the determination $^{3+}$ as $Fe(C_6H_5SeO_2)_3$ (conversion factor 0·0901) $Fe(C_{10}H_7SeO_2)_3$ (conversion factor 0·0725) by ns of ammonium benzene- (**I**) or naphthalene-inate (**II**). The limiting dilution for **I** was 3·75 × 10^5 and for **II** 1 in 6·5 × 10^5. *Gravi-c determination*—Add HNO_3 or HCl to the soln., make up to 100 ml with H_2O, introduce n. of **I** or **II** and heat the mixture for 25 to in. Filter off the ppt. on a glass filter No. 4, with H_2O, dry at 110° to 120° and weigh. The num concn. of HNO_3 (or HCl) in the test soln. *N*, and the optimum concn. of **I** and **II** are and 1·8%, respectively. The presence of Al, Ni, Cu, Be, Zn, Cd, Mn, Co, Ca, Sr or Ba not affect the determination of Fe. *Radio-c determination*—Titrate Fe^{3+} with benzene-inic acid soln. at pH 1 to 2; use ^{59}Fe as active indicator. The activity of the soln. ases during the titration to a constant value ed by the solubility of the ppt. of $Fe(C_6H_5SeO_2)_3$ × 10^{-7} mole per litre). The relative error for ation of 2 to 4 mg of Fe is ≯ ± 2%.

K. R. C.

970. **Polarographic studies with fused borax as solution. III. Polarography of iron, cobalt and nickel.** Yu. K. Delimarskiĭ and K. M. Kalabalina. *Ukr. Khim. Zhur.*, 1958, **24** (4), 435–439; *Ref. Zhur., Khim.*, 1959, (6), Abstr. No. 19,051.—The polarographic behaviour of Fe_2O_3, CoO and NiO in a basal soln. of fused $Na_2B_4O_7$ was studied and polarograms were recorded at 820°. It was shown for all three compounds that the polarograms are better satisfied by the Heyrovský - Ilkovič equation than by the Kolthoff - Lingane equation. The diffusion current is proportional to the molar concn. of the dissolved oxide. The value of the prelogarithmic coeff. (2·3 RT/nF) for Ni corresponds to a 2-electron process ($n = 2$); for Fe and Co the value of n fluctuates between 1 and 2, *e.g.*, with Ni it was shown that the temp. function of the diffusion current (i_d) is satisfied by the equation: $i_d = A(B/T)$, where T = abs. temp., whence the energy of activation can be derived. For NiO the energy of activation is 35·3 kcal. per mole. (For Part I, *cf. Anal. Abstr.*, 1958, **5**, 4003, and for Part II, *cf. Anal. Abstr.*, 1959, **6**, 1677.) K. R. C.

3971. **Successive determination of iron, copper and titanium in ferrotitanium by titration with chromous chloride.** A. I. Busev and Li Gȳn (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1959, **25** (1), 30–34.—Titration in dil. H_2SO_4 soln. with a platinum indicator electrode gives steps corresponding to reductions of Fe^{3+} and Cu^{2+}. After addition of conc. HCl and replacement of the electrode by one of mercury, a further titration at 60° to 70° gives the titanium step. A tungsten rod can also be used throughout as an electrode instead of platinum and mercury. G. S. Smith

3972. **Effect of the structure of [iron and cobalt] alloys on the results of spectrographic analysis.** I. A. Grikit. *Tekh. Ekon. Byul. Sovnarkhoz Zaporozhsk. Ekon. Adm. R.*, 1958, (2), 51–55; *Ref. Zhur., Khim.*, 1959, (5), Abstr. No. 15,054.—Spectra are produced by a condensed spark discharge, by the arc discharge of an alternating current and by an impulse discharge. To avoid oxidation of the electrodes, sparking is carried out in an atmosphere of argon. Carbon electrodes are used for the spectrographic analysis of light-alloy steels and heat-stable alloys. A more powerful spark discharge should be used in the analysis of heterogeneous heat-stable alloys. With a spark discharge, a selective "working" of the sample surface takes place, substances being mainly yielded from the grain boundaries and interdendrite spaces and from the dislocations of crystals. The effect of the structure of the alloys on the results decreases with an increase in the duration of the preliminary sparking. Eight photographs of the fine structure of the sparking zones are given. K. R. C.

3973. **Determination of impurities in ferro-niobium by spectrographic analysis.** A. B. Shaevich, Ya. M. Kalinskiĭ, N. I. Chabanenko and M. A. Perepelkina (Ural Inst. of Ferrous Metals). *Zavod. Lab.*, 1958, **24** (12), 1478–1479.—Powdered samples of ferroniobium are mixed with Fe_2O_3 and graphite (1:4:5) and pressed in a ring ≃ 0·5 mm thick on a copper or nickel disc which can be rotated at 1 r.p.m. and serves as the lower electrode. Spectra from which the contents of Al, Ti, Zr, Sn, Cu and Mn, together with lower contents of Cr, can be obtained are excited by an arc (4 amp.) with an upper carbon electrode. With high contents of Cr the dilution with Fe_2O_3 and graphite is made in the ratio 1:19:20. G. S. Smith

3974. **Spectrographic analysis of binary alloys of iron and chromium.** E. V. Lifshits, V. G. Konovalov and V. F. Erko (Phys.-Tech. Inst., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1958, **24** (12), 1483–1484.—The determination of Cr (0·1 to 30%) in iron and that of Fe (0·1 to 1%) in chromium is carried out by dissolution of the sample in dil. H_2SO_4 and the use of a Feldman porous-cup electrode.

G. S. Smith

3975. **Determination of beryllium in ferrous alloys.** S. I. Gusev and E. V. Sokolova (Perm State Medical Inst.). *Zavod. Lab.*, 1959, **25** (1), 52.—The sample of steel (0·1 to 0·3 g) is dissolved in 5 to 10 ml of conc. HCl, the soln. is evaporated to dryness, the residue is treated with 5 to 10 ml of hot water, the insol. carbides are filtered off and washed with hot water, and the filtrate (20 to 25 ml) is treated first with from 0·5 to 2·5 g of EDTA (disodium salt) and a few drops of conc. aq. NH_3, and then with 20 to 25 ml of a 2% soln. of 2-hydroxy-1-naphthaldehyde (*cf.* Gusev *et al.*, *Anal. Abstr.*, 1958, **5**, 379). The liquid is heated to $\geqslant$ 70° and treated with enough 25% aq. NH_3 to give a dark red colour and then a few drops in excess. Stirring with a glass rod gives a yellow cryst. ppt. which is filtered off after 15 min., washed with 50% ethanol, dried at 110° and weighed.

G. S. Smith

3976. **Rapid determination of copper and lead in steel and cast iron.** H. A. Nicolas (Lab. Central Sidelor-Micheville, France). *Chim. Anal.*, 1959, **41** (1), 23.—Copper and Pb are determined by electrolysis. Dissolution of the sample in HCl causes reduction of Fe to the ferrous state and addition of hydroxyammonium chloride prevents its re-oxidation at the anode. *Determination of Cu*—Dissolve the sample (5 g) in 40 ml of HCl (d = 1·19). Dilute with 50 ml of cold water and filter. Add 15 g of hydroxyammonium chloride, dilute to 200 ml, and electrolyse for 20 min. in the cold with a platinum cathode and a current of 3 amp., with continuous stirring. Wash down the beaker and the shaft of the cathode, and complete the deposition by electrolysis for a further 5 min. With steel, the copper deposit is practically pure. With cast iron, a little carbon gives it a greyish colour. Although the weight due to this is almost negligible, for precise results the deposit can be dissolved from the cathode with 5 ml of hot HNO_3 (d = 1·38); 5 ml of H_2SO_4 (1:1) is added and the mixture is diluted to 200 ml and electrolysed as before. *Determination of Cu and Pb in steel containing Pb*—Dissolution of the sample and electrolysis are carried out as described above. In the first electrolysis, Cu and Pb are deposited and weighed together. The deposit is then stripped from the cathode with HNO_3, H_2SO_4 is added, and electrolysis carried out as before. Only Cu is now deposited, and Pb is obtained by difference.

R. E. Essery

3977. **Spectrochemical determination of magnesium and the influence of the magnesium content on the analysis of silicon and manganese in cast iron.** A. S. Dem'yanchuk and E. D. Kukharenko (E. O. Paton Inst. of Electric Welding and Inst. of Machinery, Acad. of Sci., UkrSSR, Kiev). *Zhur. Anal. Khim.*, 1959, **14** (1), 45–49.—Magnesium in cast iron is determined by high-frequency excitation from the lines Mg 2802·69 and Fe 2806·98 A, with preliminary sparking for 30 sec. and exposure for 20 sec.; 0·005% of Mg may be determined. High-voltage condensed spar excitation may also be used, with preliminar sparking for 60 sec. and exposure for 45 sec. Wit these procedures Mg, Mn and Si may be determine from one photograph, with the pairs of lines M 2802·69 and Fe 2806·98; Mn 2933·1 and Fe 2926· and Si 2881·6 and Fe 2880·7 A. The presence Mg has no influence on the determination of M and Si; in an a.c. arc, however, it causes an increas in the relative intensities of the lines due to M and Si. With $\geqslant$ 0·04% of Mg in the cast iro little effect is observed; above 0·06% the resul for Mn and Si are 1·5 or 2 times the true values.

C. D. Kopkin

3978. **Determination of boron in steel and iro by the aluminium spark method.** Hong-Jang Wan (Inst. of Metal Res., Acad. Sinica). *Acta Metallur Sinica*, 1959, **4** (1), 75–80.—The sample (1 g) dissolved by heating in 70% $HClO_4$ (4 ml Tungsten is removed as trioxide by boiling wit conc. HNO_3 (2 ml), but separation of Fe is no necessary. The $HClO_4$ digest is diluted with wate and 5% NaOH soln. (25 ml) is added. Pur aluminium rods (8 mm in diameter and 5 to 10 cm in length) are used as electrodes, the ends of whic should be previously flattened and sharp edge removed. The sample soln. (0·02 to 0·05 ml) placed on the end of the electrode and evaporate to dryness at 30° to 50° with the aid of an infra-re lamp. With a controlled spark source (12,000 V electrode gap, 2·5 mm; exposure, 40 sec.), boro in the concn. range from 0·005 to 1·0% is dete mined by the line 2496·778 A with a coeff. of varia tion of $\pm$ 5·3%. Analysis of 7 samples of stee shows that the results by the proposed metho are consistently higher than those by a colori metric method.

S. H. Yuen

3979. **Quantitative spectrographic determinatio of small amounts of boron, arsenic, tellurium an phosphorus in steel.** A. S. Dem'yanchuk. *Inzh Fiz. Zhur.*, 1958, **1** (10), 88–93; *Ref. Zhur., Khim* 1959, (7), Abstr. No. 23,105.—Spectra are excite in an a.c. arc discharge from a 14-amp. generato The upper graphite electrode, which is free fro B, has a diam. of 10 mm. Calibration curves a constructed with co-ordinates of ΔS *vs.* log of the lines (in A), B 2089·59 to Fe 2087·5 As 2288·12 to Fe 2290·05, Te 2142·75 to 2145·2, and P 2149·1 to Fe 2145·2. All the elements are determined simultaneously in t following concn. ranges (in %)—B 0·001 to 0· As 0·01 to 0·30, Te 0·002 to 0·05, and P 0·010 0·12. The effect of a preliminary discharge on t intensity of the P line is studied. The slope of t calibration curve for Te is 0·33 and for As is 0· The coeff. of variation is $\pm$ 4%.

K. R. C.

3980. **Photometric determination of aluminiu in steel.** U. T. Hill (Inland Steel Co., E. Chica Ind., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 42 431.—Aluminium is determined photometrica with Eriochrome cyanine R (C.I. Mordant Blue modified by nitration. Interference by Fe suppressed either by removal as $Fe(OH)_3$ in presence of Zn and B, or by masking with ascor acid.

P. D. Parr-Richar

3981. **Determination of aluminium in constr tion steel.** E. Hekneby (Det Norske Veritas, Os *Tidsskr. Kjemi, Bergv.*, 1958, **18** (9), 148–15 The method is based on a two-stage merc cathode separation with an intermediate ammo

. and a final pptn. with cupferron. *Procedure*—olve the sample (5 g) in H_2SO_4 (1 + 9) (65 ml) filter through ashless paper (reserve the ppt. he determination of insol. aluminium). Dilute filtrate to 150 ml and carry out a mercury ode separation (2 to 2·5 hr. at 5 amp.). At end of the electrolysis add a few ml of aq. NH_3 stop the electrolysis when the gas evolution at cathode increases. Separate the electrolyte, add aq. NH_3, filter, and wash the ppt. with $_4)_2SO_4$ soln. (2%). Dissolve the ppt. in H_2SO_4 2) (5 ml), dilute to 100 ml and electrolyse n (30 min.). Oxidise the electrolysed soln. bromine water, cool, add cupferron [concn. given] and filter. Add aq. NH_3 to the filtrate ive a pH of 3 to 4, filter, wash the ppt. with water, ignite and weigh as Al_2O_3. The method aimed to give satisfactory results for aluminium ents > 0·005% on 5-g samples.

B. Rönnholm

3982. **Spectrographic determination of carbon gh-alloy steels.** A. S. Dem'yanchuk. *Inzh.-Fiz.*, 1958, **1** (9), 116–118; *Ref. Zhur., Khim.*, , (7), Abstr. No. 23,074.—With large amounts i in steel, the nickel line (2296·5 A to 2297·1 A) the carbon line (2296·9 A) overlap The spectro-hic determination of C in steel is then only ble with high dispersion. By using a mag-m electrode at the beginning of spark excitation e spectra the vapour is enriched with carbon a simultaneous decrease in the concn. of Ni. phenomenon is utilised in the spectrographic rmination of C. Spectra are excited in a spark arge or in a high-voltage condensed spark arge by a complex method (current 3 amp., city 0·02 μF, control spark gap 1·5 mm, tical spark gap 1 mm). The end of the esium electrode is cylindrical with a diam. 3 mm. During exposure the sample is either ed continuously or twice subjected to a 5-sec. in sparking. Calibration curves are con-ced of Δ *S vs.* log *C*. The coeff. of variation determination is $\pm$ 5%. K. R. C.

3983. **Determination of titanium, zirconium, m and tantalum in steels: separations by exchange.** J. L. Hague and L. A. Machlan Bur. of Standards, Washington, D.C., U.S.A.). *s. Nat. Bur. Stand.*, 1959, **62** (1), 11–19.—ample (5 to 25 g) is dissolved in warm dil. and Ti, Zr, Nb and Ta are concentrated and ted from the bulk of Mn, Cr, Ni, Co and Fe ective pptn. with cupferron at 5°. The ppt. tted to the oxides which, after treatment with and HF to remove SiO_2, are fused with O_7, and the melt is dissolved in a mixture l (50%, v/v) and HF (10%, v/v). The soln. sed through a short anion-exchange column x 1, ≃ 200 mesh), after which four separate s containing Ti + Zr, Mo + Sn + Fe ded), Nb, and Ta are obtained by successive with (*i*) HCl - HF, (*ii*) HCl - HF - NH_4Cl, H_4Cl - HF and (*iv*) NH_4Cl - NH_4F adjusted 5 to 6. Boric acid is then added to the three to complex the fluoride and the elements td. with cupferron at 5° (Fe is added as for Ti and Zr, and Zr as carrier for Nb and After removing V and W by pptn. with soln., Ti can be determined by differential photometry of the H_2O_2 complex and Zr etrically as ZrP_2O_7 (+ HfP_2O_7). Niobium a can be determined gravimetrically as or spectrophotometrically by the quinol and pyrogallol methods, respectively. The accuracy is satisfactory over the range of 0·001 to 0·5% of each element, except for Zr in concn. < 0·01%. A complete analysis requires several days, but the method compares favourably in time and convenience with alternative procedures.

W. J. Baker

3984. **The spectrographic determination of phosphorus in steels and welded joints.** E. S. Kudelya. *Avtomat Svarka*, 1958, (9), 88–92; *Ref. Zhur., Khim.*, 1959, (7), Abstr. No. 23,078.—By means of the radio-isotope ^{32}P it is shown that on the excitation of spectra in a spark or arc discharge a rapid consumption of P arises from the low diffusion rate of P in steel. In order to raise the diffusion rate of P it is recommended that the current be raised to 14 amp. Combustion curves show that in the initial period (15 sec.) there is a sharp decrease in the intensity of the P line (2149 A) and then a very slow decrease for nearly 3 min. Phosphorus has a max. intensity with a stationary carbon electrode; the rate of combustion in this case is minimal. Spectrograms in the case of the a.c. arc discharge are taken without a condenser on photographic plates, type III, at a distance of 180 mm from the light source to the aperture and exposure time of 35 to 45 sec. Calibration curves are constructed of Δ *S vs.* log *C*; the comparison line is the 2145·20-A line of Fe. The minimum determinable concn. of P is 0·006 to 0·008%; the error is 3 to 7%. The method is suitable for the localised analysis of steel and welds. K. R. C.

3985. **Coulometric determination of oxygen in steel by the fusion - extraction process in a stream of argon.** K. Abresch and H. Lemm (Chem. Hauptlab. d. August Thysen-Hütte A.-G., Duisburg-Hamborn). *Arch. Eisenhüttenw.*, 1959, **30** (1), 1–6.—The conventional fusion - extraction process is modified by de-gassing the sample in a stream of argon instead of under vacuum. The CO evolved is oxidised to CO_2 and titrated coulometrically. The time required for the determination is 7 min. (0·5 g of sample) and the method gives a standard deviation of 0·0012%. H. Sawistowski

3986. **Effect of carbon on the spectrochemical analysis of chromium steel.** Kung-Soo Chang and I-Djen Ho (Inst. of Metal Res., Acad. Sinica). *Acta Metallurg. Sinica*, 1959, **4** (1), 87–91.—The spectrochemical determination of Cr in steel is found to be affected by the content of C of the sample. The error becomes significant when the concn. of C is > 1%. Experiments show that C affects the volatilisation of various elements from solid electrodes and the effect on Cr is greater than on Fe. S. H. Yuen

3987. **Carbonate method for the determination of micro amounts of uranium in iron.** I. E. Starik and F. E. Apollonova. *Trudy Komiss. Anal. Khim.*, 1958, **9** (12), 264–273; *Ref. Zhur., Khim.*, 1959, (8), Abstr. No. 26,990.—The radio-isotope ^{233}U is used to study the separation of U (10^{-5} to 10^{-8} g) from Fe; Fe^{3+} are pptd. with alkali-metal carbonates and the U in soln. is converted into a carbonate complex. The concn. of the ammonium salt affects the degree of pptn. of $Fe(OH)_3$; the amount of Fe^{3+} remaining in soln. rises noticeably with an increase in the concn. of ammonium salts > 15% and ammonium carbonate > 1%. The greatest solubility of $Fe(OH)_3$ is observed with

K_2CO_3 and the least with ammonium carbonate and $NaHCO_3$. The pptn. of Fe^{3+} takes place in a narrow pH range (for ammonium carbonate, 7·5 to 7·6). At pH 3 to 3·5 and $> 7·0$, U is hardly adsorbed at all. The amount of U adsorbed also decreases with an increase in the CO_3^{2-} concn.; with a single separation of U and Fe in a 1% carbonate soln. $\simeq$ 3% of U is adsorbed on $Fe(OH)_3$; micro amounts of U are completely separated from Fe if the pptn. is repeated. When separating small amounts of U (10^{-6} to 10^{-8} g), losses are observed owing to adsorption of U on glass, and on platinum vessels during evaporation of the soln. to dryness. Such losses can be almost eliminated if co-pptn. is carried out with metatitanic acid, the removal of the ammonium salt by sublimation being omitted. The results obtained indicate that the carbonate method can be used for the separation of 10^{-6} to 10^{-8} g of U from 10^5 times as much Fe.
K. R. C.

3988. **Comparative evaluation of different methods for the spectrographic determination of nickel in steel.** É. I. Braĭnin (Stalin Metallurgical Works). *Zavod. Lab.*, 1958, **24** (12), 1479–1483.—The merits of six different methods in use in the USSR for determining Ni (0·2 to 4·8%) in steel are discussed.
G. S. Smith

3989. **Determination of cobalt by precipitation chromatography.** R. N. Golovatŷĭ and V. V. Oshchapovskiĭ. *Ukr. Khim. Zhur.*, 1958, **24** (4), 491–494; *Ref. Zhur., Khim.*, 1959, (7), Abstr. No. 23,062.—2-Nitroso-1-naphthol (**I**) was used for the determination of Co^{2+} in a column of adsorbent. In the detection of Co in soln. of its salts **I** was used alone and also in admixture with the anionite VS (1:1) or chromatographic Al_2O_3 (1:1). At pH 6 to 7 the detectable minimum was 0·06 μg of Co in 1 drop of soln. Ferrous iron was first oxidised to Fe^{III} which was then masked with pyrophosphate. By this method 1·5 μg of Co in a soln. containing cations of the alkaline-earth elements, Al^{3+}, Fe^{3+}, Mn^{2+}, Zn^{2+}, Ni^{2+}, Pb^{2+}, Cu^{2+} and Cd^{2+} (0·3 *N* concn. of each ion) could be detected. A 1000-fold excess of Mo or V did not affect the analysis if the chromatograms were washed with 0·1 *N* HCl. In the chromatography of Co and Ni from a single soln., a mixed zone was obtained from which Ni was eluted with 0·1 *N* HCl. In the quant. determination of Co in the presence of Ni, the anionite VS (impregnated with **I**) was used as the adsorbent. The test soln. (0·1 ml) and the standard soln. (0·1ml) were introduced into two separate columns which were both washed with 0·1 *N* HCl until the Ni was completely removed (1 to 2 ml) and then with H_2O. The Co - **I** complex was then eluted with 10 to 20 ml of ethanol. To the eluate were added 10% citric acid soln. (3 ml) and conc. aq. NH_3 soln. (5 ml), and the mixture was made up to 45 ml; to this was added an alkaline soln. of **I** (3 ml) and the soln. was made up to 50 ml and measured colorimetrically.
K. R. C.

3990. **Determination of cobalt in metallic nickel.** B. E. Reznik, R. E. Dlugach and R. T. Rudskaya (Dniepropetrovsk State Univ.). *Zavod. Lab.*, 1958, **24** (12), 1431–1432.—The complex of nitroso-R salt with Co should be formed at a pH of $\simeq$ 8 to 8·5, but the measurement of extinction is best carried out in acid medium since at low pH strict regulation of pH is unnecessary. The sample (0·2 g) of metallic nickel is dissolved in 10 ml of dil. HNO_3 (1:1), oxides of N are boiled off, a small amount of ZnO together with 10 ml of Na acetate soln. (concn. not stated) are added, and the boilin soln. is treated with 10 ml of a 0·1% soln. of nitroso R salt. After 1 min. the ppt. is dissolved by th addition of 10 ml of dil. HNO_3 (1:1), the coole soln. is diluted to 50 ml and the extinction i measured. With contents of Co between 0·2 an 1·2%, the method is accurate to $\pm$ 0·02% of th sample wt.
G. S. Smith

3991. **Photometric determination of nickel in th solutions for production of electrolytic zinc.** S. D Djordjević (Zav. za Fiz. Kem. i Elektrokem Belgrade, Yugoslavia). *Bull. Soc. Chim., Belgrad* 1957, **22** (5–7), 377–382.—A procedure for th determination of Ni in the soln. used for com mercial zinc electrolysis has been developed an tested. A 150-ml flask containing 40 ml of 25% NaOH soln. is heated and 5 ml of fresh aq. K_2S_2O soln. (4%, w/v) is added, followed by 10 ml o sample. The $K_2S_2O_8$ prevents the pptn. of Zn(OH) while $Ni(OH)_2$ is pptd. together with the hydroxide of other impurities. The mixture is filtered an the ppt. is dissolved in HCl (1:1). Nickel i extracted with $CHCl_3$ and determined photo metrically as the dimethylglyoxime complex. blank is necessary for all determinations. Neithe Mn nor other impurities in the sample affects th accuracy of the method. Comparative results ar given.
A. Grochowski

3992. **Adsorption of ruthenium from aqueou solutions on ion-exchange resins.** E. I. Il'enko B. P. Nikol'skiĭ and A. M. Trofimov. *Trud Komiss. Anal. Khim.*, 1958, **9** (12), 148–160; *Re Zhur., Khim.*, 1959, (8), Abstr. No. 27,067.—Th conditions of adsorption of the nitrosonitrat complexes of Ru from aq. soln. on to ion-exchang resins were studied by means of the radio-isotop ^{106}Ru.
K. R. C.

3993. **Separation and determination of rhodiu and iridium.** E. Jackson (The Sheffield Smeltin Co. Ltd., Royds Mill St., Sheffield, England *Analyst*, 1959, **84**, 106–110.—The soln. of th metal chlorides is heated to fuming with HN and H_2SO_4 containing Li_2SO_4 and heating is co tinued after addition of $HClO_4$. The diluted dige is treated with thioacetanilide in glacial acet acid and then with $CrCl_2$ soln. The collecte washed ppt. of a rhodium complex is ignite reduced in hydrogen, evaporated with HF, tran ferred to filter-paper with HCl and washed, ignite reduced, heated to 650° to 700° in a current of and treated with dil. aqua regia; the collected pp is ignited, reduced to metal and weighed. T filtrate containing the iridium is evaporated wi HNO_3 and H_2SO_4, heated to 250° and oxidis with HNO_3 and $HClO_4$. Solid thiourea is add and the ppt. of iridium sulphide is collecte ignited, reduced to metal and weighed. V small amounts of iridium sulphide may be fum with HNO_3 and H_2SO_4 - Li_2SO_4 and then w HNO_3 and $HClO_4$; the filtered soln. is evaporat then oxidised and the Ir determined by titrati with standard quinol soln. in the presence of 3: dichlorobenzidine (Pollard, *Bull. Inst. Min. M* 1948, **67**, 9). A modification of the method for determination of Ir in refined rhodium sponge described. Copper, Hg, Ag, Au, Pd, Os, Pt, Ru, Re and W interfere.
A. O. Jones

3994. **Amperometric titration of iridium w the rotating platinum electrode.** N. K. Pshenit and N. A. Ezerskaya (Inst. of Gen. and Ind Chem., Acad. of Sci., USSR, Moscow). *Zh*

l. Khim., 1959, **14** (1), 81–86.—Small amounts r (10^{-5} to 10^{-3} *M*) are titrated amperometrically n quinol (**I**) or ascorbic acid (**II**). At room temp. educes Ir^{IV} very rapidly at the platinum elec- le; the titration is carried out in the presence HCl (pH 1·5) in 0·1 *N* NaCl; a change of pH in range 0·3 to 3·0 has no effect on the deter- ation. For $1·5 \times 10^{-3}$ to 1×10^{-5} *M* Ir^{IV}, average error is 1 to 2%. No interference is sed by the presence of Rh, Pt, Pd, Cu, Ni, Se Te; Au^{III} interferes. In the presence of Ru^{IV}, aqua regia to the soln., evaporate to dryness n HCl in the presence of NaCl to complex Ru, ch in 25-fold excess does not then interfere. ric iron is complexed with H_3PO_4; 100-fold ounts do not interfere. At room temp. **II** uces Ir^{IV} stoicheiometrically at the platinum trode, in the acidity range 10^{-3} to 10 *N* HCl; 5 to 10 *N* the rate of reduction is decreased. relative error in determining 10^{-5} to 5×10^{-3} Ir^{IV} is 1 to 5%; with $< 10^{-5}$ *M* the error may 10 to 15%. The determinable minimum is 2 mg of Ir in 10 ml. No interference is caused the presence of Rh, Pt, Pd, Cu, Ni, Pb, Se or Au, Ru and Fe interfere. The interference of (50-fold amounts) is removed as described above that of Fe^{III} with H_3PO_4. C. D. Kopkin

995. **Photometric determination of platinum in presence of silver.** V. F. Barkovskiĭ. *Izv. sh. Uchebn. Zavedeniĭ. Khim. i Khim. Tekhnol.*, 8, (3), 52–55; *Ref. Zhur., Khim.*, 1959, (7), tr. No. 23,067.—*Procedure* (*i*)—Treat the silver - inum alloy (0·1 g) with HNO_3 (1 : 2) (5 ml), ove the oxides of N, filter, wash the platinum lue with hot H_2O acidified with HNO_3 and olve in aqua regia by heating. Dilute the HNO_3 ate containing the bulk of the Ag to 35 to 40 ml H_2O, add conc. HCl (0·5 ml), heat to complete coagulation of AgCl, cool, filter and wash the l ppt. with H_2O. After separation of AgCl add filtrate with the washings to the platinum soln., porate twice almost to dryness with HCl and with H_2O and add H_2O (50 ml) to the residue; to the soln. (1 ml) a 1% aq. soln. of thiosemi- azide (**I**) (1 ml), make up to 25 ml with borate er soln. (pH 9), stir, and after 35 min. measure ometrically with a red filter in a 5-cm cell, nst H_2O. The relative error is $\gtrsim 2·5\%$, and letermination takes 13 to 15 hr. as compared 30 hr. for the gravimetric determination of *Procedure* (*ii*)—Heat the alloy (0·05 g) with O_3 (1 : 1) (2 ml) until the Ag is completely lved, add HCl (1 : 1) (1 ml) and heat until the as completely dissolved. Evaporate the soln. the ppt. almost to dryness, add H_2O (3 to 5 ml) 20% NH_4SCN soln. (20 ml), stir to complete lution of AgCl, then add aq. NH_3 soln. (1 : 1) the colour changes from orange - red to w; then add 20% Na acetate soln. (30 ml) make up to 100 ml with H_2O. To a 10-ml ot add 20% NH_4SCN soln. (5 ml), 0·5% in soln. (3 ml) and 1% **I** soln. (5 ml), stir, 20% Na acetate soln. (20 ml), stir, set aside hr., make up to 100 ml with H_2O, shake and measure photometrically with a green in a 3-cm cell. The time required for a single mination is 2 to 2·5 hr. and the absolute error 0·29%. K. R. C.

96. **Application of the thermobalance for the itative analysis of clays.** A. Grizo and M. azić-Stevanović (Zav. za Neorgan. Kem. ol., Belgrade, Yugoslavia). *Bull. Soc. Chim., Belgrade*, 1957, **22** (5–7), 383–393.—Thermogravimetric methods for the analysis of the mineral composition of clays were investigated on a series of Yugoslav clays and imported kaolins. The samples were sieved to $< 88\ \mu$ and their dehydration losses on ignition above 300° were used for the determination of the kaolinic substance. A comparative table was compiled with the results obtained by chemical analysis, Berdel and Kallauner-Matejka rational analysis, X-ray, differential thermal analysis (DTA) and thermogravimetric analysis (TGA). Good agreement was obtained between TGA, DTA and X-ray methods, but the chemical and rational analyses gave less satisfactory results. A. Grochowski

3997. **Relation of infra-red spectra and chemical analysis for some chlorites and related minerals.** W. M. Tuddenham and R. J. P. Lyon (Kennecott Res. Center, Salt Lake City, Utah, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 377–380.—By relating the i.r. spectra to the chemical analysis of a series of 21 chlorites and related minerals, both the degree of substitution of Al for Si (*Y* number) and the total Fe content of an unknown mineral sample can be estimated from i.r. data. The structural type can also be deduced readily from the same data. K. A. Proctor

3998. **Rapid analysis of glasses and raw materials using EDTA.** R. Sales (Brit. Glass Ind. Res. Ass., Sheffield, England). *J. Soc. Glass Tech.*, 1959, **43**, 37T–53T.—Full details are given of the use of EDTA in the titrimetric determination of (*i*) CaO in limestone (indicator, murexide + naphthol green B), (*ii*) CaO and MgO in colourless glasses [Patton and Reeder indicator (*cf. Anal. Abstr.*, 1956, **3**, 2982)] and (*iii*) PbO and Al_2O_3 in lead crystal glasses (titration with Zn acetate in the presence of EDTA, with Xylenol orange as indicator). J. A. Sugden

3999. **Analysis of soda - lime - silica batches and its application to glassworks investigations.** S. M. Budd (United Glass Bottle Manufacturers Ltd.). *J. Soc. Glass Tech.*, 1959, **43**, 54T–58T.—Sodium carbonate and $CaCO_3$ were determined in the same 1-g sample of batch from which the cullet had been removed by sieving a 2-lb bulk sample through a 5 B.S. sieve. *Procedure*—After standing for 30 min. with 50 ml of H_2O in a 250-ml flask, the sample was titrated with 0·2 *N* HCl (thymol blue, pH 9). This titre gave the amount of acid required to convert the Na_2CO_3 into $NaHCO_3$ without hydrolysis of the $CaCO_3$; 5 ml of 50% HCl soln. was added to the titrated soln. and after 30 min. titration was begun with EDTA (disodium salt) (25 g per litre). After the addition of 12 ml, the soln. was made alkaline with a 2-ml excess of KOH soln. (20%) and 0·2 g of murexide powder (prepared by grinding 0·2 g of murexide with 42 g of K_2SO_4), and the $CaCO_3$ was titrated to the change from violet to blue. Data are recorded to illustrate how, by planning of the experiments and statistical treatment of the results, it is possible to estimate errors arising from sampling, analysis, and batch heterogeneity. J. A. Sugden

4000. **Application of the flame photometer and the spectrograph to the analysis of a soda - lime glass.** R. J. Powell and J. Todd (Gen. Electric Co., Wembley, England). *J. Soc. Glass Tech.*, 1959, **43**, 73T–84T.—The determination of the alkali

metals and CaO by flame photometry and of CaO, Al_2O_3 and MgO by spectrography is described in detail. Flame photometry was successful for the alkali metals if care was taken to make the standards and samples as alike as possible. The results for CaO in SGT Glass No. 1 were somewhat lower than the officially published figure. The spectrograph also gave satisfactory results, and preliminary trials with solid samples in place of soln. gave no better reproducibility.

J. A. SUGDEN

4001. **Determination of the sodium, potassium and lithium oxide contents of soda - lime - magnesia, soda - boric oxide - silica, and potassium oxide - lead oxide - silica glasses using an EEL filter flame photometer.** W. W. Fletcher (Brit. Glass Ind. Res. Ass., Sheffield, England). *J. Soc. Glass Tech.*, 1959, **43**, 86T–93T.—Full details are given. The instrument is suitable for routine control analyses. J. A. SUGDEN

See also Abstracts—**3860**, **3861**, Indicators for EDTA titrations. **3862**, Mo^{III} as volumetric reducing agent. **3864**, Radiometric titration with ^{60}Co complexes. **3865**, Radioactive isotopes in anionite separations. **4191**, Boron in soil and plants. **4192**, Aluminium in soil and plants. **4212**, Qual. separation of metals.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

4002. **Determination of carbon and hydrogen in organic compounds. I. Efficiency of combustion catalysts.** J. Horáček and J. Körbl (Inst. of Chem., Acad. of Sci., Prague, Czechoslovakia). *Mikrochim. Acta*, 1959, (2), 303–313 (in German).—A study of the oxidation catalytic activity of 28 catalysts used as combustion catalysts in ultimate analysis has been made and the practical value of these substances is discussed. The most effective is the decomposition product of $AgMnO_4$.

B. B. BAUMINGER

4003. **Elementary organic analysis by the "wet combustion" method.** A. P. Terent'ev and B. M. Luskina (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1959, **14** (1), 112–117.—The method is carried out by heating the sample at 150° to 160° with a mixture of conc. H_2SO_4 and a conc. aq. soln. of chromic acid (10 : 1 or 2) in a stream of O (50 to 60 ml per min.). Pass the gaseous reaction products through a train consisting of a tube containing Cr_2O_3 on pumice heated at 700° to 750°, a gas bubbler containing glass pieces moistened with a soln. of hydrazine hydrate neutralised to pH 6 with acetic acid, a similar bubbler moistened with conc. H_2SO_4, a tube of anhydrone, and a tube of anhydrone and ascarite. The max. error in determining C was 0·5%. Chlorine forms CrO_2Cl_2, which is decomposed by the Cr_2O_3 to liberate Cl and O or by water vapour to form HCl; Br-containing compounds decompose to form Br. The halogens are absorbed in the hydrazine hydrate soln., CO_2 passing through for absorption with ascarite; the halogens are det mined by Volhard's method. To determine N compounds that form NH_3 when heated in co H_2SO_4, use the Kjeldahl method on the residu soln. from the combustion. Metals remain in t oxidation residue as sulphates. After reduci Cr^{VI} to Cr^{III} with a few drops of methanol, det mine Fe by adding Na_2S, and after evolution H_2S titrate Fe^{2+} with $K_2Cr_2O_7$ soln. Determine iodimetrically in the sulphate soln.; N can then determined by the Kjeldahl method. Silico containing compounds are decomposed to for silicic acid; add 2% gelatin soln., filter, ignite a weigh. C. D. KOPKIN

4004. **Rapid methods of micro-elementa analysis. XVI. Dependence of the length contact layer on the method of decomposition in t direct determination of oxygen.** M. O. Korsh and E. A. Bondarevskaya (Inst. of Elemento-organ Compounds, Acad. of Sci., USSR, Moscow). *Zh Anal. Khim.*, 1959, **14** (1), 123–127.—Prelimina pyrolysis of an oxygen-containing organic substan in a quartz tube inside a quartz combustion tube 900° to 1000° in pure dry N liberates up to 95 of the O as H_2O, CO and CO_2; passage over a lay of C at 1150° converts these and other produc into CO, which is oxidised with CuO to CO_2; th is then absorbed with ascarite and weighed. T preliminary pyrolysis enables the contact layer C to be considerably shortened (*e.g.*, to 5 c instead of 16 to 20 cm). The carbon layer may inside the quartz tube above the sample; by slow moving the heater from the open towards t closed end of the tube, O is quant. converted in CO. (*Cf. Korshun et al., Anal. Abstr.*, 1959, 2625.) C. D. KOPKIN

4005. **Rapid micro-determination of organical bound halogens, arsenic, phosphorus and boro** M. Corner (Nat. Chem. Lab., Teddington, Midd England). *Analyst*, 1959, **84**, 41–46.—The meth described is an extension of the flask combusti method of Schöniger (*cf. Anal. Abstr.*, 1955, 1816; 1957, **4**, 924). In the determination of A HCl, hydrazine dihydrochloride and KBr are intr duced into the flask after combustion, and As is distilled into $NaHCO_3$ soln., which is th titrated with standard iodine soln. For Cl, t products of combustion are absorbed in wat which is then titrated with standard $AgNO_3$ so The products of combustion of compounds c taining P are absorbed in water, the soln. evaporated with a drop of HNO_3 and the P pptd. as quinoline molybdophosphate. This collected, dissolved in standard NaOH soln. a determined by back-titration. In the determinati of B, the sample is mixed with finely powde KOH before combustion, the products are collec in water, and the B is determined under the app priate conditions by titration in the presence mannitol after removal of CO_2 by means of inert gas. A. O. JONE

4006. **New methods for the quantitative det mination of halogens in organic substances.** P. N. Fedoseev and M. Ya. Sobko (S. O. Maka Nikolaev Shipbuilding Inst.). *Zhur. Anal. Khi* 1959, **14** (1), 118–122.—For the simultane determination of Cl and Br or Cl and I in orga substances containing N, ignite the sample (to 0·1 g) at 750° to 800° in a quartz tube in a stre of O (200 to 250 ml per min.), pass the prod successively through a layer of Cr_2O_3 at 800°

0°, and a tube of KBr or NaBr at 600° to 700°; ss the exit gases successively through a tube ntaining powdered cryst. Na_2CO_3 moistened with % NaOH and H_2O_2 and a tube containing 10% OH and H_2O_2 soln. After the ignition wash out ese two tubes with water, add 3 or 4 ml of conc. NO_3, boil for 3 to 5 min., cool, add 20 ml of *N* $AgNO_3$ and 2 or 3 ml of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ d titrate with KSCN to determine total halo- ns. Chlorine is calculated from the change in . of the KBr or NaBr tube. To determine logens in compounds containing N and S, omit e bromide tube and titrate the halogen absorbed the alkaline peroxide. The error of the deter- nations is ± 0·3%. (*Cf. Anal. Abstr.*, 1959, **6**, 30.)

C. D. Kopkin

4007. **Analysis of organofluorine compounds. nultaneous micro-determination of fluorine and rogen in organic compounds.** N. É. Gel'man, O. Korshun, M. N. Chumachenko and N. I. rina (Inst. Elem. Org. Comp., Acad. Sci. USSR, scow). *Dokl. Akad. Nauk SSSR*, 1958, **123** (3), 3–470.—A sample (3 to 8 mg) is placed in a ca test-tube and covered with a granulated xture of NiO with 15 to 20% of MgO. The test- oe is then put in a silica-lined furnace and colytic combustion in a CO_2 atmosphere is rried out at 900° to 950°. Samples of liquids are tt put into thick-walled quartz capillary tubes, ich are inserted into test-tubes and covered h an oxidiser. On completion of the com- tion and displacement of the evolved N into measuring tube, the residue is transferred to a ohydrolysis tube (*cf.* Korshun *et al.*, *Anal. tr.*, 1958, **5**, 1542), through which steam at 0° and at a rate of 0·5 ml of condensate per . is passed for 20 to 25 min. The F^- are then ated in the condensate with $Th(NO_3)_4$ soln. Korshun *et al.*, *Anal. Abstr.*, 1956, **3**, 2112) the vol. of N is measured. Both platinum and cel tubes are suitable for the pyrohydrolysis, when the nickel tube is used, empirical correc- s are applied for losses of F in the apparatus ng the operation. The accuracy of the method ·2% absolute for N, and 0·5% for F. A table calculated and determined F and N contents for mber of organofluorine compounds is given.

A. Grochowski

008. **Determination of small amounts of organic thur in small samples.** J. P. Dixon (Shell Res. , Thornton, Chester, England). *Chem. & Ind.*, , (5), 156.—In the determination of 10 to μg of organic S in samples of < 25 mg by aation of Ag_2SO_4 on silver gauze, followed by tion of the Ag_2SO_4 soln. with KI, greater racy can be attained by using (*i*) the rapid oustion procedure of Ingram, and (*ii*) electro- silver as prepared by Mitsui and Sato (*Mikro- . Acta*, 1956, 1603). To ensure a minimum tant blank, the silver gauze should be cooled before removal of the Ag_2SO_4 with boiling olated distilled water. Chlorine interferes, but ne absence of S it can be determined by this edure if an ammoniacal wash soln. replaces the al hot-water wash. The excess of ammonia is ved and, although AgCl is then pptd., it ars to be titrated quant. by the KI soln.

W. J. Baker

009. **Determination of microgram quantities of ur by reduction with Raney nickel.** L. Granatelli [American Oil Co. (Texas), Texas City, U.S.A.]. *Anal. Chem.*, 1959, **31** (3), 434–436.—The sample (minimum S content 5 μg) in *iso*-propyl alcohol is caused to react under N with Raney nickel. After desulphurisation for 30 min., HCl is added dropwise and H_2S is purged from the system with N and absorbed in a mixture (1:1) of *N* NaOH and acetone (previously swept through with N) and a little dithizone in acetone. A few drops of standard Hg acetate soln. are added to the absorbent as the addition of acid to the sample begins; when all the acid is added, the sample is boiled under reflux and the H_2S liberated is titrated with Hg acetate until the absorbent remains pink. The N flow is discontinued and the absorbent allowed to suck back a little way up the delivery tube to recover adsorbed H_2S. Several different types of S compounds were analysed; consistent recoveries of ≃ 95% were obtained. Olefins (> 2%) cause appreciable error.

P. D. Parr-Richard

4010. **Semi-micro determination of boron in organic compounds.** J. A. Kuck and E. C. Grim (Micro-anal. Lab., Stamford Res. Lab., American Cyanamid Co., Conn., U.S.A.). *Microchem. J.*, 1959, **3** (1), 35–41.—A sample (0·1 to 0·2 g) is covered with 10 to 15 drops of liquid paraffin and burnt in a Parr high-pressure oxygen bomb containing 25 ml of water, and the soln. is made up to 100 ml with water. An aliquot (10 ml) is brought to pH 4·8 (pH meter) with 0·01 *N* HCl and heated under reflux for 10 min. (air condenser) to remove CO_2. While the soln. is cooling a stream of pre-purified N is passed through by means of a filter-stick. The stick is washed down with water, the pH adjusted to 5·15 and 10 ml of 10% aq. mannitol soln. added. The mixture is titrated with 0·05 *N* NaOH, a pH meter and glass electrode being used, with magnetic stirring. The exact end-point (pH 7·5 to 8·0) is located by the method of differencing (Kolthoff, "pH and Electro Titrations," Wiley, N.Y., 1931, pp. 120–122). Boron (%) = $100(VNF)/W$, where V is the vol. (ml) of NaOH, N its normality, F the factor applicable to mannitol titrations (10·82) and W the wt. (mg) of sample. Results of analyses of 10 compounds (1·69% to 19·71% of B) showed good agreement with theoretical values. Replicate analyses showed a relative accuracy of 0·4%.

R. E. Essery

4011. **Non-aqueous determination of acetylenic hydrogen.** L. Barnes, jun. (Air Reduction Co. Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 405–408.—The method is based on the liberation of H^+ when $AgClO_4$ reacts with mono-substituted acetylenic compounds. The acid is titrated with a methanolic soln. of tri(hydroxymethyl)methylamine, with screened thymol blue as indicator. The method can be applied in the presence of certain weak organic acids without pre-neutralisation; strong acids and bases may be pre-neutralised to prevent interference. Acids and amines of intermediate strength (K_A 10^{-4} to 10^{-1} and K_B 10^{-9} to 10^{-12}) interfere owing to buffer action; halogens and aldehydes do not interfere. Recoveries are generally > 99·6%.

G. P. Cook

4012. **Direct titration of hydroxyl groups with lithium aluminium di-*n*-butyl amide.** L. A. Small (Arnold, Hoffman & Co. Inc., 55 Canal St., Providence, R.I., U.S.A.). *Analyst*, 1959, **84**, 117–119.

Lithium aluminium di-*n*-butyl amide is sufficiently strong a base to titrate hydroxyl hydrogen as an acid in ether solvents (*cf.* Higuchi *et al.*, *Anal. Chem.*, 1952, **24**, 685). In the original method an excess of the reagent in tetrahydrofuran was added, the excess being then back-titrated with standard butyl alcohol - xylene soln. With some ethanolamines the addition of an excess of reagent caused difficulties and a method of direct titration was developed in which the titrant is a 0·25 *N* soln. in dimethoxyethane. This soln. can be dispensed from a burette with a Teflon stopcock. The titration is carried out in an atmosphere of N, the sample is dissolved in tetrahydrofuran and the indicator is a soln. of 4-phenylazodiphenylamine in benzene. The apparatus used is described. The precision of the method is better than 1%.

A. O. Jones

4013. **Detection of thiocarbonyl groups by infra-red spectroscopy.** E. Spinner (Australian Nat. Univ., Canberra). *J. Org. Chem.*, 1958, **23** (12), 2037–2038.—The applicability of i.r. spectroscopy to studies of thiol - thione equilibria was examined and conjugated C:S groups were found to give rise to absorption bands as intense as carbonyl stretching bands. The i.r. spectra of N-methylpyridine-2-thione and N-methylpyridine-4-thione were compared with those of the corresponding carbonyl compounds. Strong absorption in the range 1110 to 1145 cm^{-1} was observed in the spectra of the thio compounds, while the keto compounds displayed only comparatively weak absorption in this region ($\epsilon \not> 60$). No other band characteristic of the C:S group, and absent from the spectra of the C:O derivatives, has been found.

K. R. C.

4014. **Vapour-phase chromatography of light hydrocarbons and inert gases (carbon dioxide, carbon monoxide, nitrogen, oxygen, hydrogen).** J. Ronbaut and C. Fodderie (Raffinerie "Esso Belgium", Anvers). *Ind. Chim. Belge*, 1958, **23** (8), 845–854 (in French).—The light hydrocarbons (C_1 to C_4) and CO_2 are separated by gas - liquid chromatography; the "inert" gases are analysed on a column of molecular sieve. For the first stage, the carrier and sample are dried and H_2S is removed from the sample by a column of MnO_2. The conditions are—column length, 8 metres; temp., 30°; stationary phase, tetrabutylene (30% by wt.) on 30 to 60-mesh firebrick treated with HNO_3; carrier, He at a flow rate of 75 ml per min. and pressure of 20 p.s.i.g.; sample size, 1 ml. Before the sample enters the second column it is dried, H_2S and CO_2 are removed by a soda-asbestos filter and the hydrocarbons, except CH_4, are removed by a column of activated charcoal. The column is packed with Linde Molecular Sieve 5A of controlled moisture content and operated at 30° with He carrier flowing at 42 ml per min. All the components give separate peaks except *n*- and *iso*-butylenes.

G. Burger

4015. **Analysis of light hydrocarbon gas mixtures.** J. A. Barnard and H. W. D. Hughes (Dept. of Chem. Engng, Univ. Coll., London, England). *Nature*, 1959, **183**, 250.—Olefins are separated from saturated compounds, and C_2 gases from the C_3 ones on a single column containing a physical mixture of the packings previously used separately (Bradford *et al.*, *J. Inst. Petrol.*, 1955, **41**, 80; Bednas and Russell, *Canad. Chem. J.*, 1958, **36**, 1272). It is also possible to separate *iso*buten from these gases on the same column.

H. F. W. Kirkpatrick

4016. **Effect of various factors on the chromato graphic separation of hydrocarbons in the gaseou phase. I. Effect of quantitative composition o the separation of mixtures of methane and ethane** D. A. Vyakhirev and A. I. Bryk. *Uch. Zap Gor'kovsk. Inst.*, 1958, (32), 43–53; *Ref. Zhur. Khim.*, 1959, (6), Abstr. No. 19,173.—The colum was filled with silica gel, grain size 0·2 to 0·4 mm (height of the column 41 cm, cross-section 3·9 sq cm, temp. 15° to 16°); the carrier gas was air a a rate of flow of 30 ml per min. The concn. of effluen from the column was measured with a Zeiss inter ferometer. It was shown that an increase i ethane concn. of the mixture impairs the separa tion whereas an increase in methane concn. doe not affect it. With very small methane concn and very large ethane concn., activated carbo AG-2 is the most suitable adsorbent.

K. R. C.

4017. **Determination of *cis*- and *trans*-2:3-epoxypentane.** D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959 **31** (7), 1281.—Infra-red data are given.

4018. **Determination of hydroxyl concentratio in polypropylene glycols by infra-red spectroscopy** E. A. Burns and R. F. Muraca (Calif. Inst. Technol. Pasadena, U.S.A.). *Anal. Chem.*, 1959, **31** (3) 397–399.—The –OH stretching vibration at 2·84 μ of 14 polypropylene glycols follows the Lambert Beer - Bouger law with a coeff. of variation o ± 2·2%, and a precision (99% confidence limits of 0·273 ± 0·0047 g per millimole. The effectiv molar absorptivity is calculated as 6·74 g per milli mole per mm at a slit width of 0·012 mm.

G. P. Cook

4019. **Zinc complex of toluene-3:4-dithiol as reagent for ketose sugars.** R. E. D. Clark an R. G. Neville (Boeing Airplane Co., Seattl Wash., U.S.A.). *J. Org. Chem.*, 1959, **24** (1), 110 112.—*Procedure for sugar analysis*—To dil. HC (0·25 ml) add 0·3 to 0·5 mg of sugar with the sam amount of the zinc complex, and heat the mixture t boiling for 30 to 60 sec. In the presence of ketose the liquid begins to turn yellow within a fe seconds, with the deposition of an orange - yello ppt. within 0·5 to 2·0 min., depending upon th concn. This method was compared with other the anthrone reagent of Johanson (*cf. Anal. Abst* 1954, **1**, 502), was found to be inferior to the dithio taking seven times as long for the colour to develo fully. The final colour intensity and sensitivi appeared to be of the same order of magnitude f both methods.

K. R. C.

4020. **Nuclear magnetic resonance to distingui between aliphatic aldehyde and ketone derivative** D. Y. Curtin, J. A. Gourse, W. H. Richardson a K. L. Rinehart, jun. (Noyes Chem. Lab., Uni Ill., U.S.A.). *J. Org. Chem.*, 1959, **24** (1), 93–97. The nuclear magnetic resonance spectra of 2: dinitrophenylhydrazones (**I**) and semicarbazon (**II**) were calibrated in c.p.s. at 40 Mc relative H_2O as zero. Samples were spun in a precisi annular cell and an external sample of CH (− 104 c.p.s.), benzene (− 76 c.p.s.) or H (0 c.p.s.) was used for the calibration point

spectrum. A high-resolution nuclear magnetic nance spectrometer with a 12-in. magnet and stabiliser was used to determine the spectra. io-frequency side-bands were generated with an io-oscillator. Chloromethane was used as the ent for both **I** and **II**. The concn. of soln. of **I** e 20% (w/v), with the following exceptions—aldehyde (9%), propionaldehyde (13%) and ptaldehyde (7%). The concn. of soln. of **II** e—*iso*butyraldehyde 20%, α-ethylbutyralde-e 15%, *n*-amyl methyl ketone 7%, di-*n*-butyl ne 14% and pinacolone 10%. The proton nance of the N=C–H group of the chloro-hane soln. of **I** occurs between — 109 and 18 c.p.s., and the corresponding resonance uency of **II** is between — 86 and — 97 c.p.s. ne compounds studied. K. R. C.

021. **Determination of 2:2-dichloropropionic , 2-chloropropionic acid and 2:2:3-trichloro-ionic acid in chlorinated propionic acid by use mercuric salts.** R. P. Marquardt and E. N. e (Dow Chem. Co., Midland, Mich., U.S.A.). *l. Chem.*, 1959, **31** (3), 418–421.—The methods the determination of 2-chloro- and 2:2-dichloro-ionic acids are based on their conversion into ıvic acid, which reacts with $Hg(NO_3)_2$ to form -bis(hydroxymercuri) - 3 - nitratomercuripyruvic . This compound reacts with KI to form KOH, h is measured titrimetrically. The 2:2:3-loropropionic acid method is based on the ction by this acid of mercury propionate in ng aq. soln. to precipitate a mixture of curous salts, which react with KOH to produce). After dissolving the oxide in dil. acid, the is oxidised and is titrated with standard SCN soln. Each acid can be determined in the ence of the others with an accuracy of ± 0·5% e absolute value. G. P. Cook

022. **Gas - liquid chromatography of fatty atives. I. Separation of homologous series of fins, *n*-hydrocarbons, *n*-nitriles and *n*-alcohols.** E. Link, H. M. Hickman and R. A. Morrissette ner-Daniels-Midland Co., Minneapolis, U.S.A.). *mer. Oil Chem. Soc.*, 1959, **36** (1), 20–23.—itions have been established for the successful ration of 1-olefins from C_8 to C_{24}, saturated drocarbons from C_1 to C_{24}, fatty nitriles from o C_{22}, and fatty alcohols, including those ned by the reduction of the fatty acids from aut, soya-bean and castor oils. Separation of mixed methyl esters of hydrogenated fatty of castor oil has also been effected. A station-hase of Apiezon L is used for hydrocarbons, owax 400 monostearate and Carbowax for ols and nitriles, and silicone (Wilkens No. 1) ohols, with a 30 to 60-mesh firebrick support. m is the carrier gas and the column and tor are operated at 220° to 260°. Peaks are fied by their retention vol. and by the method ernal standards, and peak areas are measured a compensating planimeter.

G. R. Whalley

023. **Estimation of methacrylic esters with ric acetate by non-aqueous titration.** K. L. and M. N. Das (Jadavpur Univ., Calcutta, *Chem. & Ind.*, 1959, (5), 162.—Methyl and methacrylates can be determined by the -catalysed reaction between a known excess 1 *M* mercuric acetate and the sample in nol at 45° for ≃ 30 min. Thymol blue is added to the reaction mixture, followed by a nearly satd. soln. of NaCl in ethanediol until the indicator colour changes to yellow. The soln. is then titrated with 0·1 *N* HCl in butanol until the colour changes sharply from yellow to pink. The amount of unsaturation is obtained from the difference between the two titrations of mercuric acetate before and after the reaction. The error is ≃ ± 0·1% for the methyl ester and ⩾ + 2% for the ethyl ester. W. J. Baker

4024. **Determination of acrylonitrile [vinyl cyanide] in various materials.** J. M. Štěpánek, V. M. Černá and V. J. Patková (The Inst. of Hygiene, Srobarova, Prague). *Analyst*, 1959, **84**, 65–66.—The sample of water (500 ml) or aq. suspension of ≃ 250 g of foodstuff, grain, etc., containing 0·5 to 100 mg of vinyl cyanide is mixed with *iso*propyl alcohol (25 ml) and conc. H_2SO_4 (5 ml). The mixture is saturated with NaCl, pieces of porous porcelain and glass are added and the liquid is slowly distilled in an apparatus of specified dimensions into a receiver containing 0·1 g of thiourea and 0·15 ml of conc. HCl per ml of distillate expected. For 0·5 to 20 mg of vinyl cyanide, 2 ml is distilled; for larger amounts, 5 to 10 ml. Distillation of the azeotropic mixture takes place at ≃ 78°. The distillate is treated by the method previously described (Štěpánek and Černá, *Analyst*, 1958, **83**, 345). A. O. Jones

4025. **Refractometric method of determining caprolactam and N-methylcaprolactam.** A. S. Shpital'nyĭ and L. A. Vol'f (S. M. Kirov Leningrad Textile Inst.). *Zavod. Lab.*, 1958, **24** (12), 1489.—The contents are obtained by measuring $n_D^{18°}$ and by the use of a calibration graph.

G. S. Smith

4026. **Electrometric determination of some thiols and disulphides.** R. Willemart and P. Fabre (Serv. de Rech., Roussel-Uclaf, Paris-Romainville, France). *Ann. Pharm. Franç.*, 1958, **16** (11), 676–678.—Thiols (6 Br per –SH group) and disulphides (10 Br per –S·S– group) can be titrated with bromate in the presence of bromide. The thiol alone can be titrated with bromate in the presence of iodide. The end-point is indicated by the change of potential across platinum electrodes at constant current (arrangement described). *Procedure*—The mixture (0·01 mole per 100 ml) is dissolved in water, 25 ml is acidified with 15 ml of *N* HCl, and 0·5 g of KI is added. The soln. is titrated with 0·1 *N* $KBrO_3$ at a constant current of 25 micro-amp. To 5 ml of the original soln. are added 25 ml of water, 10 ml of conc. HCl and 0·5 g of KBr. The soln. is titrated in the same way with 0·1 *N* $KBrO_3$. If 0·01 *N* $KBrO_3$ is used, the constant current must be ≃ 1·5 micro-amp. Examples are given of titrations of mixtures of thioglycollic and dithiodiglycollic acids and of cystine and cysteine. E. J. H. Birch

4027. **Determination of isomeric thiolacetates in binary mixtures.** D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (6), 1120.—Infra-red data are given.

4028. **Colorimetric micro-determination of arsenic in primary lewisite.** R.-M. Fournier. *Mém. Poudres*, 1958, **40**, 385–388.—*Procedure*—Mineralise organic arsenic by boiling under reflux with aq. $NaHCO_3$ soln.; oxidise to As^{5+} by the addition of an aq. soln. of iodine in KI; convert into molybdoarsenate and reduce to molybdenum

blue by boiling under reflux for 5 min. with an H_2SO_4 soln. of ammonium molybdate and hydrazine sulphate. The method is very sensitive (3 μg of lewisite per ml corresponding to 0·35 μg of As per ml) and rapid (15 min.). K. R. C.

4029. **Determination of non-aromatics in high-purity C8-alkylbenzenes.** S. H. Hastings and D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (7), 1281.—Infra-red data are given for C_7 to C_9 olefins, paraffins and cycloparaffins.

4030. **Determination of C9- to C15-*iso*propylbenzenes.** D. E. Nicholson and B. H. Johnson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (7), 1281.—Infra-red data are given.

4031. **Determination of diphenyls related to 1:3:5-trimethylbenzene.** D. E. Nicholson (Humble Oil and Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (6), 1120.—Infra-red data are given.

4032. **Separation of *m*-xylene and *p*-xylene by gas chromatography.** D. H. Desty, A. Goldup and W. T. Swanton (British Petroleum Co. Ltd., Sunbury on Thames, England). *Nature*, 1959, **183**, 107–108.—7:8-Benzoquinoline gives a better separation of the xylene isomers than any other stationary phase previously reported. The isomers are completely resolved at 78·5° on a column 12 metres long with an internal diameter of 0·25 mm, with N (42 ml per min.) as carrier gas. Retention-vol. data are listed. G. P. Cook

4033. **Determination of α-(4-chloro-2-methylphenoxy)propionic acid in commercial acid of this name.** L. A. Haddock and L. G. Phillips (I.C.I. Ltd., Gen. Chemicals Div., Res. Dept., Widnes Lab., Lancs., England). *Analyst*, 1959, **84**, 94–101.—By the method described, α-(4-chloro-2-methylphenoxy)propionic acid (**I**) is separated chromatographically from α-(4:6-dichloro-2-methylphenoxy)propionic acid, α-(6-chloro-2-methylphenoxy)propionic acid and α-(2-methylphenoxy)-propionic acid. An ethereal soln. of the acids extracted from the sample is applied to a standardised chromatographic column prepared by a specified procedure with a static phase of Hyflo Super-Cel and buffer soln. of pH 7·3, and a moving phase of diethyl ether - $CHCl_3$ (1 : 1), and the eluate is divided into three main fractions. Under the conditions described, the extinction of the second fraction at 287 mμ is due entirely to **I**, which can thus be measured by reference to a calibration graph. Certain β-phenoxypropionic acids present in some commercial samples have absorption spectra similar to those of the α-analogues, but these do not interfere since they are eluted before any of the α-compounds. The method is reliable for samples of the mixed acids containing at least 75% of the active constituent.

A. O. Jones

4034. **Analysis of butoxyethoxypropyl esters of 2:4-dichlorophenoxyacetic acid and 2:4:5-trichlorophenoxyacetic acid mixtures.** F. J. Witmer, D. N. Thomas and J. B. Vernetti (Chipman Chem. Co., Portland, Ore.). *Anal. Chem.*, 1959, **31** (7), 1280.—Infra-red data are given.

4035. **Analysis of 2:4-dichlorophenoxyacetic acid *iso*propyl ester and 2:4-dichlorophenoxyacetic acid butyl ester mixtures.** F. J. Witmer, D. N. Thomas and J. B. Vernetti (Chipman Chem. Co., Portland Ore.). *Anal. Chem.*, 1959, **31** (7), 1280.—Infra-re data are given.

4036. **Determination of phthalide in produc from oxidation of o-xylene.** D. E. Nicholso (Humble Oil and Refining Co., Baytown, Tex. *Anal. Chem.*, 1959, **31** (7), 1281.—Infra-red da are given.

4037. **Polarographic determination of meth salicylate.** V. D. Bezuglÿĭ and V. N. Dmitriev (Kharkhov Dental Materials Works). *Zavod. Lab* 1958, **24** (12), 1446–1447.—The sample (0·5 g *e.g.*, of a mixture containing 58·9% of phenoco hydrochloride, 26·7% of eucalyptus oil and 14·4% of methyl salicylate, is dissolved in 10 ml methanol, and a 0·5-ml aliquot is mixed wit 4·5 ml of a basal electrolyte of 0·05 *N* tetramethy ammonium iodide in a mixture of methanol an water (4:1). After passage of H, the polarographi wave ($E_{\frac{1}{2}}$ = − 2·02 V) is recorded and measured The reduction is irreversible. The relation betwee wave height and concn. is linear. G. S. Smith

4038. **Chromatographic separation of mixture of nitro- and dinitro-benzoic acids.** K. A. Oglobli and G. V. Markina. *Vestn. Leningr. Univ.*, 195 No. 4, *Ser. Fiz. i Khim.*, (1), 149–153.—Aluminiu oxide containing 5 to 17% of H_2O and tinted wit aq. methyl orange (1% soln.) was the adsorben Benzene or a 1:1 mixture of benzene with dieth ether or acetone was used as solvent. The sampl soln. containing 0·1 g of each acid in 10 to 25 m of solvent was poured into the column. After th bulk of the soln. had been taken up under the usu pressure, a developer was added and the passag of the liquid through the adsorbent was facilitate by the application of a low pressure. The adsorbe acids were recovered by treatment with a mixtu of benzene and dil. HCl. The separation of pai of the following acids was attempted— *o*-, *m* and *p*-nitrobenzoic acids [(**I**), (**II**) and (**III**)], 2: dinitro- and 3:5-dinitro-benzoic acids [(**IV**) and (**V**) Complete separation was obtained for **I** and **II** **II** and **III**, **II** and **IV**, **III** and **IV**, and **IV** and **V** **II** was the only acid separated in a pure conditio from mixtures of **I** and **II** and of **II** and **V**.

K. R. C.

4039. **Improved method for the paper-chromat graphic separation of dinitrophenylamino alcoho** W. Grassmann and A. Reidel (Max-Planck-In f. Eiweiss-u.-Lederforsch., München). *Hoppe-Se Z.*, 1958, **312** (4–6), 206–209.—Paper chromat graphy of dinitrophenylamino alcohols and relat compounds may be carried out on ether-wash paper with a 12·5% (w/v) soln. of thymol in NaOH. The migration relationships found in t solvents usually employed are reversed so that t results obtained correspond to those obtained reversed-phase chromatography. Two-dimensio separations are possible in combination with o of the common solvent mixtures. A procedure intensifying the colour of the spots is described.

F. Powell

4040. **Simultaneous spectrophotometric det mination of toluene-o- and -p-sulphonamides their commercial products.** K. Kámen and Oplištil (Spolana n.p., Neratovice, Czechoslovaki *Chem. Průmysl*, 1959, **9** (1), 14–17.—The met is based on the different absorptions of toluen sulphonamide (248·0 mμ) and its *p*-isor

'4·4 mμ) in NaOH soln. The extinctions, which additive, follow the Beer - Lambert law over a de range if the soln. are protected against CO_2. e proportions of both components can be termined by means of a mathematical relation ived from extinctions of the pure compounds. *ocedure*—Dissolve the sample (0·2 g), dried at 5°, in NaOH soln. (4%) (50 ml) and dilute to itre with H_2O. After 1 hr. measure the extinc- ns at 248·0 and 274·4 mμ. The error in the termination of the major component in the xture is $\simeq \pm 2\%$. When brown samples are alysed, a preliminary decolorisation must be rried out. Dissolve the sample (2 g) in ethanol %), shake with charcoal, dilute to 100 ml, er and evaporate 10 ml to dryness. Dissolve the idue in NaOH soln. (4%) (50 ml) and proceed described above. J. ZÝKA

4041. **Aspects of gas chromatography and selective lrotreatments.** R. M. Soemantri. *Proefschr. h. Hogesch. Delft*, 1958, 1–80 (in English).— s - liquid partition chromatography procedures given for the quant. analysis of mixtures of phthalene with dekalin and tetralin, mixtures of henyl, di*cyclo*hexyl- and *cyclo*hexyl-benzene, l of mixtures of all six compounds. The separa- n is effected in a coiled copper tube (180 to cm × 4, 6 or 12 mm) packed with either ctyl phthalate on crushed firebrick or silicone on Celite; operating temp. are from 170° to °, the carrier gas is N and thermal-conductivity ection is used. The sample is 0·15 to 0·20 ml nzene soln.). The method of calibration described ased on correction factors and can be applied to ary mixtures of bicyclic compounds as well as multicomponent mixtures. The max. error for n component (after correction) is $\simeq \pm 1{\cdot}5\%$; minimum determinable concn. is $\simeq 1\%$.
W. J. BAKER

042. **Detection of the ferrocene nucleus in a plex reaction mixture.** S. I. Goldberg (Wright- terson Air Force Base, Ohio, U.S.A.). *Anal. n.*, 1959, **31** (3), 486.—The method is based on destruction of the ferrocene (di*cyclo*penta- yl iron) nucleus by oxidation of the Fe^{2+} to Fe^{3+} with H_2O_2; the Fe^{3+} are detected with SCN^-. The ferrocene is separated from complex tion-mixtures by paper chromatography, with zene as the developer. G. P. COOK

043. **Determination of sulphur in coal products he method of double combustion.** V. I. Dal', A. Zakupra and I. N. Ruban (Dniepropetrovsk n. Tech. Inst.). *Zavod. Lab.*, 1958, **24** (12), 1445–1446.—The method of Volȳnskiĭ and dakova (*cf. Anal. Abstr.*, 1956, **3**, 2110) is plified and shortened by the use as absorbent n aq. soln. of iodine containing starch.
G. S. SMITH

044. **Determination of linalol: a comparative y of the Glichitch and Fiore methods by gas - id partition chromatography.** D. Holness ever Ltd., Cent. Perfumery Dept., 1, South- Park Road, London). *Analyst*, 1959, **84**, .—The reaction products of the Glichitch o-formic) and the Fiore (acetyl chloride - thylaniline) methods for the determination of ol and other tertiary alcohols in essential oils examined by gas - liquid chromatography and bearing on the reliability of analytical results scussed. The products of the Fiore reaction with linalol are much the simpler, the chief product being linalyl acetate, with traces of unchanged linalol and its anhydrides. The Glichitch reaction is complex, with linalyl formate as the main product and considerable amounts of linalyl acetate, the formates and acetates of three other alcohols and smaller amounts of unchanged linalol and its anhydrides. There are more potential sources of error in the Glichitch method, and when the sources of error revealed by gas - liquid chromatography are combined, results by the Fiore method would appear to be $\simeq 1\%$ lower and those by the Glichitch method 3 to 5% lower than the true linalol content. Both methods give reproducible results and also consistent patterns in gas - liquid chromatograms. The preference for the Glichitch method of the Essential Oils Sub-Committee of the Analytical Methods Committee of the Society for Analytical Chemistry (*Analyst*, 1957, **82**, 325) needs reviewing since the results, although reproducible, are consistently biased. A. O. JONES

4045. **An improved determination of the cineole content of eucalyptus oils.** E. Klinger. *Riechstoffe u. Aromen*, 1959, **9** (1), 5–7.—A rapid and reliable m.p. method is described. *Procedure*—A 10-ml sample is placed in a test-tube which passes down through the bung of a vacuum flask and dips into diethyl ether contained in a small vessel resting on the bottom of the flask. The sides and bottom of the tube must be surrounded by ether, and preferably a rubber ring surrounds the tube at the top of the ether vessel to slow down evaporation of the ether in subsequent steps. A thermometer (range − 30° to + 20°, graduated by 0·1°) is placed in the sample, and vacuum is applied to the flask to evaporate the ether and cool the sample, which is stirred occasionally to prevent supercooling. When the sample freezes the temp. is noted; the vacuum is then shut off and the sample allowed to re-melt, the temp. being measured when the last crystals disappear; 1 or 2 repeat freezings and re-meltings are made by restoring and cutting off the vacuum. The figures are averaged and the content of cineole (**I**) is read off from a graph of m.p. *vs.* content. Ordinary commercial oils can be analysed directly; those of lower **I** content (< 60%) can be analysed by making the determination after adding a known amount of pure **I**.
H. L. WHITEHEAD

4046. **Moisture-measuring apparatus [for use with materials of high resistivity, such as paper, textile fabrics, wood veneers, etc.].** Isotope Products, Ltd. [Inventor: J. A. H. Hart]. Brit. Pat. 814,395; date appl. 10.10.55.—The apparatus comprises a pair of spaced electrodes adapted to make electrical contact with the material being tested, a source of alternating voltage of low frequency (20 to 200 c/s), means for supplying a signal voltage as a result of the voltage drop in current flow, and means for amplifying the signal, and for filtering out and measuring the resistive components of the current flow. J. M. JACOBS

4047. **Volumetric determination of chloride in paper and water by a polarisation end-point procedure.** D. Price and F. R. Coe (Res. Dept., I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Analyst*, 1959, **84**, 62–64.—The apparatus is that previously described (*Analyst*, 1959, **84**, 55). For the determination of Cl^- in paper and similar materials, a 20-g sample of the

material (cut into 1-in. squares) is boiled with water for 30 min., the cooled extract is filtered and the combined filtrate and washings are diluted to 500 ml. For water, a 100-ml aliquot is acidified with 10 ml of glacial acetic acid. The liquid is then placed in the circuit in the amperometric apparatus (*loc. cit.*) and is titrated from a microburette with 0·01 *N* $AgNO_3$. Near the end-point, galvanometer readings are taken after each 0·1-ml addition of titrant. At the end-point the titration graph reaches a minimum value and with excess of titrant reverses its direction. A. O. Jones

4048. **Quality of fillings for bedding, upholstery, etc. Part 2: Curled hair and curled hair/fibre mixtures, loose or layered.** British Standards Institution (2 Park St., London, W.1). B.S. 2894: Part 2:1958. Amendment No. 1, 20.4.1959.—Amendments to the method of carrying out the cleanliness test are described.

4049. **Polarographic determination of the cooking degree of pulp.** F. Dubšík (Severočeské Papírny, Štětí, Czechoslovakia). *Papír a Celulosa*, 1959, **14** (1), 5–7.—The Künge method for the determination of the cooking degree of pulp is based on the oxidation of lignin with $KMnO_4$, and has been modified so that the unused $KMnO_4$ is determined polarographically by means of the wave of Fe^{2+} in citrate buffer soln. after its reaction with the excess of $KMnO_4$. The interference of Mn^{2+} formed during the reduction is avoided by carrying out a blank. J. Zýka

4050. **Improved spent sulphite liquor determination by the nitrosolignin method.** O. Goldschmid and L. F. Maranville (Rayonier Inc., Shelton, Wash., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 370–374.—The Pearl and Benson method (*Paper Trade J.*, 1940, **111**, 235) was improved by a filter combination giving more nearly monochromatic light of approx. 430 mμ, which eliminated undesirable absorption due to the nitrite reagent and improved sensitivity and reproducibility. Interference from certain substances can be partly eliminated by preparing a blank soln. from the sample at the same pH as the reagent-treated sample. The precision (3 σ) for a single determination is ± 0·15 and ± 0·75 p.p.m. at the 1 and 5 p.p.m. levels, respectively, from 5 determinations at each level. G. P. Cook

4051. **Determination of the water content of polyamide melts with the Karl Fischer reagent by the dead-stop method.** G. Glöckner and W. Meyer (Thuringsche Kunstfaser Werk "Wilhelm Peck", Schwarza). *Faserforsch. u. Textiltech.*, 1959, **10** (2), 83–84.—In a rapid method for determining the water content of polyamide melts (finally dehydrated melts or melts in the course of their dehydration), the sample is extracted with anhyd. methanol and the product is titrated with a Karl Fischer reagent to an end-point which is determined electrically, either by the polarisation current (dead stop) or the polarisation voltage method. The presence of momomer, lactam, etc., in the melt does not affect the accuracy of the results. H. L. Whitehead

4052. **Determination of bound styrene in raw and cured polymers by nitration.** C. L. Hilton, J. E. Newell and J. Tolsma (U.S. Rubber Co. Research Center, Wayne, N.J.). *Anal. Chem.*, 1959, **31** (5), 915–917.—The sample of polystyrene copolymer is boiled with HNO_3 under reflux. Aq. NaOH soln is added, leaving the soln. still strongly acid. The product, mainly *p*-nitrobenzoic acid, is extracted with diethyl ether and extracted from the ether soln., with aq. NaOH soln. The extinction is measured at 265 mμ, 273·75 mμ and 288 mμ, and the styrene content calculated. The extinction of the nitration products of polystyrene and of the other component of the copolymer must be determined on the instrument in use. Lack of agreement at the three wavelengths indicates interferences which invalidate the method for that sample. Such interferences may be caused by materials that yield similar nitration products. Most compounding ingredients are removed by a preliminary extraction with $CHCl_3$, and the remainder, which interfere with other methods, do not interfere. The accuracy and precision are within 1% (relative). G. Burger

4053. **New spot test for epoxy resins.** M. H. Swann (U.S. Army Ordnance, Aberdeen Proving Ground, Md.). *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1958, **30**, 1277–1279.—Two drops of resin soln (or equiv. of dry resin) are dissolved in a few ml of conc. H_2SO_4 and the soln. is diluted with conc. H_2SO_4 until the colour intensity is similar to that of 0·1 *N* $K_2Cr_2O_7$. A drop or two of the soln. is then streaked across a horizontal acid-washed filter paper; if bisphenol-type epoxy resins are present a bright purple colour develops in 1 min. which eventually turns blue. The test is specific for epoxy resins; bisphenol - formaldehyde ester gums do not give a positive test, and only conc. acid soln. of oils and resins containing conjugated unsaturation would give interfering colours. A sensitivity to 1% of epoxy resin in coating material may be obtained, but this is reduced in the presence of products darkening in conc. acid soln. To distinguish unreacted epoxy resins from their fatty acid esters, the material, dissolved in 2-(2-ethoxyethoxy)ethanol, is treated with lepidine; the unreacted resins give a blue colour. L. A. O'Neill

4054. **Simple method for the detection of silicones.** G. von Finck (Farbenfabrik. Bayer, A.-G.). *Melliand Textilber., Eng. Ed.*, 1959, **40** (1), 32.—*Procedure*—Add conc. H_2SO_4 (1 to 1·5 ml) to the textile (50 to 100 mg), and heat with continuous shaking, withdrawing the test-tube from the flame every 3 to 5 sec. to make observations. If, after shaking, the acid runs down the inside of the tube and leaves it evenly wetted the result is negative, but if the acid runs down at very high speed like water down a metal surface the result is positive. The effect disappears when the H_2SO_4 begins to boil, or after the acid has boiled for some time according to the amount of the silicone present. Amounts of 0·5 to 1 mg of a polysiloxane can be detected by this method; silicic acid esters do not give the reaction. K. R. C.

See also Abstracts—**3897**, Ba and SO_4^{2-} in detergents. **3926**, Tin in organic compounds. **39**[illegible] Sulphur in organic compounds. **4189**, Naphthenic acid in natural waters. **4207**, Improvements in Kjeldahl method. **4208**, Fractionation apparatus.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

055. **System of clinical chemical analysis.** B. n (Vet. Admin. Hosp., Bronx, N.Y., U.S.A.). . *Chem.*, 1959, **5** (1), 62–70.—Glucose, urea N, Na and K are determined in the filtrate from ml of serum after deproteinising with $Ba(OH)_2$ - O_4. H. F. W. KIRKPATRICK

056. **Carbon-14 respiration pattern analyser for ical studies.** B. M. Talbot, M. Kirk and F. ıam (Radiation Lab., Univ. of Calif., Berkeley). . *Sci. Instrum.*, 1959, **30** (2), 116–120.—The gn and operational details are given of an rument for measuring the total ^{14}C per unit of e, CO_2 and ^{14}C specific activity, *i.e.*, ^{14}C as per- tage of C_2, in the breath of human subjects. apparatus comprises an ionisation chamber - rating reed electrometer combination for ^{14}C rmination, an i.r. CO_2 gas analyser and a bination recorder - ratio analyser.
G. SKIRROW

057. **Report of bioassay procedures [for radio- vity in urine].** W. A. Brobst (Chicago Operations ce, Health and Safety Div., AEC). *U.S. mic Energy Comm.*, Rep. COO–213, 1957, pp.—Various procedures for the analysis of e for radioactivity are presented. Details are ıded for Pu, Th and U. A list is included of pment, with approx. cost.
NUCL. SCI. ABSTR.

058. **Characterisation of complex mixtures of ıral substances by simultaneous multi- ensional paper chromatography.** P. Decker and Kikodse (Physiol. Inst., Tierärztl. Hochschule, over, Germany). *Z. anal. Chem.*, 1959, **165** (5), -343.—Four-dimensional paper chromatography six combinations of four solvents is used in the stigation of aromatic and heterocyclic substances athological urines. A scheme in which six two- ensional paper chromatograms are used is trated. B. B. BAUMINGER

059. **New method of determining the specific ity of very small quantities of urine.** L. aszewski and B. Czartoryska (Hosp.-Clinic 3, Acad. of Med., Warsaw, Poland). *Clin. r. Acta*, 1959, **4** (2), 297–301 (in French).— erol soln. are prepared with sp. gr. covering range 1·001 to 1·025. To each is added a few cals of Na benzoate and a little methylene A drop of glycerol soln. is added to a column he urine (3 to 4 cm in height and approx. m in diameter) about 0·5 cm below the surface. sp. gr. of the urine is that of the glycerol soln., drop of which remains suspended. If necessary urine may be diluted with water.
H. F. W. KIRKPATRICK

060. **X-ray fluorescence (spectroscopy) as a for the analysis of sub-microgram quantities e elements in biological systems.** S. Natelson S. L. Bender (Biochem. Lab., Roosevelt Hosp., York, U.S.A.). *Microchem. J.*, 1959, **3** (1), 4.—After an exposition of the fundamental iples of this non-destructive method of analysis, the types of instruments employed, and their advantages and disadvantages for various determinations are detailed, and the method is illustrated by a full discussion of the assay of thyroxine in blood serum by the determination of iodine. The method is especially useful for determining the metals in organic compounds during production, and for the determination of P, S and one or more halogens in organic compounds. Biological materials such as blood serum can readily be assayed for Ca, P, S, K and Cl, but present instruments will not allow of the determination of C, H, O or N.
R. E. ESSERY

4061. **Determination of copper in blood.** S. T. Fetisova. *Trudy Stalinabadsk. Med. Inst.*, 1957, (23), 195–198; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (8), Abstr. No. 9285.—Two methods of determining Cu were examined. The sensitivity of the diphenylcarbazone method was 0·1 μg, but that of the dithizone method was too low for the analysis of blood. K. R. C.

4062. **Rapid micro-quantitative method for estimating serum calcium.** T. A. Harper (Group Path. Lab., City Gen. Hosp., Sheffield, England). *Nature*, 1959, **183**, 232–233.—Add the serum (0·1 ml) to H_2O (4·9 ml), then add piperidine (1 drop) and satd. aq. murexide (centrifuged before use) (0·25 ml). Mix and read the extinction at 500 mμ. Run a standard soln. (0·1 ml) containing 9 mg of Ca per 100 ml and a water blank concurrently and calculate the concn. of Ca in the test serum. A correction for this method for correlation with the oxalate pptn. method is obtained by analysis of commercially available standard analysed freeze-dried serum.
H. F. W. KIRKPATRICK

4063. **Determination of calcium and magnesium in blood using one indicator.** H. M. C. Robinson and J. C. Rathbun (War Memorial Children's Hosp., London, Ont., Canada). *Canad. J. Biochem. Physiol.*, 1959, **37** (2), 225–229.—Calcium and magnesium together are titrated with EDTA from a micrometer-syringe burette, with Eriochrome black T as indicator, the end-point being determined photometrically; Ca is removed by pptn. as oxalate and the Mg is determined alone on the filtrate by a similar titration.
H. F. W. KIRKPATRICK

4064. **Micro-determination of calcium and magnesium in blood serum and cerebrospinal fluid.** G. Hunter (Cowley Rd. Hospital, Oxford). *Analyst*, 1959, **84**, 24–28.—The apparatus used has been previously described (*Analyst*, 1958, **83**, 93). The sample is de-proteinised with acetic acid (cerebrospinal fluid is heated in boiling water). Calcium is determined as previously described (*loc. cit.*), an equal vol. of alkaline murexide soln. being placed in each cup of the colour comparator. The ammeter is adjusted to zero, 0·5 ml of protein-free serum filtrate or 1 ml of cerebrospinal fluid filtrate is placed in the right-hand cup and standard Ca soln. is added to the other cup until the ammeter again registers zero. For the determination of Mg and Ca together, the procedure is similar, with buffered Eriochrome black T soln. in each cup. The sample filtrate (0·2 ml) is added to the right-hand cup and the liquid in the other cup is titrated with standard Mg soln. The Mg content is then ascertained from a calibration graph. End-points can be determined with an accuracy of ± 0·1 μg

of Ca and ± 0·01 μg of Mg. The inherent error is ± 2% for Ca and ± 2·5% for Mg. The trace metals of serum may cause slight interference, particularly with Mg determinations, in which an error of + 2·5% caused by Cu or Zn is probable.
A. O. JONES

4065. **Determination of silicic acid in biological substrates.** O. M. Gulina. *Material. Vopr. Prom. Toksikol. Klin. Prof. Bolez.* (*Gorkii*), 1957, 109–123; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (7), Abstr. No. 7855.—The basis of this colorimetric method is the reaction of silicic acid with ammonium molybdate, which not only removes phosphate ions and pigments but also renders the determinations more accurate. The optimum pH values for the coloured molybdosilicic acid soln. were 1·4 to 1·9. The error of determination for blood was 5% and for urine 10%. K. R. C.

4066. **Determination of iodine-131 in urine.** J. E. Marriott (Anal. Dept., Medical Div., A.E.R.E., Harwell, Berks., England). *Analyst*, 1959, **84**, 33–37.—To a 900-ml aliquot of a 24-hr. sample are added 20 mg of KI (as carrier), 125 ml of conc. H_2SO_4 and, gradually, 25 g of $KMnO_4$. After 1 hr. the mixture is heated under reflux for 4 hr. with an activated carbon gas-trap surmounting the condenser (to retain Cl). Oxalic acid is added, at 60°, and the liquid is distilled, until the vapour temp. reaches 120°, into a litre bottle containing CCl_4 connected to a Dreschel bottle containing aq. NaOH soln. The distillates in the litre bottle and the NaOH soln. are treated separately with HNO_3 and $NaNO_2$ and are extracted separately with CCl_4. The combined extracts are washed with water, the iodine is extracted with water containing a little SO_2, the aq. extract is then made alkaline and boiled to remove SO_2, and finally acidified with HNO_3. The iodide is pptd. with $AgNO_3$ soln., the ppt. is washed and collected by centrifuging, and counted with a suitably calibrated β-counter. Quoted results indicate a recovery of 75·1 to 82%.
A. O. JONES

4067. **Modification of the spectrophotometric determination of carbon monoxide in blood enabling direct determination of per cent. haemoglobin saturation.** H. Berninger and R. Smith (10th Gen. Med. Lab., APO 180, New York, U.S.A.). *Clin. Chem.*, 1959, **5** (2), 127–134.—The method of Harper (*J. Lab. Clin. Med.*, 1952, **40**, 634) is modified by inclusion of an appropriate reagent blank so that the final reading is equivalent to the total haemoglobin. H. F. W. KIRKPATRICK

4068. **Rapid micro-method for determining carbon tetrachloride in blood.** A. C. Kondos and G. L. McClymont (Fac. of Rural Sci., Univ. of New England, Armidale, N.S.W., Australia). *Analyst*, 1959, **84**, 67–68.—The reaction used is that of Fujiwara. A 3-ml portion of heparinised blood is shaken with acetone (7 ml) and after a few minutes is shaken again and then centrifuged. A 2-ml portion of the clear liquid is added to 7 ml of pyridine (purified, if necessary, by re-distillation) and, after mixing, 20% (w/v) NaOH soln. (3·5 ml) is added. A suitable glass marble is placed in the mouth of the test-tube, which is then heated in boiling water for exactly 2·5 min. An aliquot of the coloured pyridine layer is removed, 1·5 ml of abs. ethanol is added and the extinction is measured at 535 mμ and referred to a calibration graph, which is linear up to 175 μg of CCl_4 per ml. At higher concn., dilution with abs. ethanol is neces sary. A standard CCl_4 - blood soln. of approx equal concn. should be examined simultaneously especially at extremely low concn. of CCl_4. Th recovery is 95%, and the lowest limit of sensitivit is 4 μg per ml. A. O. JONES

4069. **Determination of barbiturates by automati differential spectrophotometry.** L. A. William and B. Zak (Office of the Wayne County Examiner Detroit, Mich., U.S.A.). *Clin. Chim. Acta*, 1959 **4** (2), 170–174.—The use of the Beckman DK-2 automatic ratio-recording spectrophotometer is described. H. F. W. KIRKPATRICK

4070. **Determination of dithizone in blood.** R. I Veksler (Gorky Med. Inst., USSR). *Biokhimiya* 1959, **24** (1), 42–45.—A 0·5% aq. NH_3 soln. was added to dithizone in such an amount that the dithizone concn. did not exceed 10 mg per ml of final soln. After 10 min. in a water bath at 60° to 70°, the warm soln. was filtered and the warm filtrate mixed with blood. Water was used instead of blood in the controls. Dithizone soln. was added to defibrinated blood at 37° in the *in vitro* tests or injected into a vein in the *in vivo* tests. The dithizone-containing defibrinated blood (2 ml) was extracted in a horizontal position with $CHCl_3$ (5 ml) for 15 min. at 130 vibrations per min. The extinction of the $CHCl_3$ layer was measured at 619 mμ. The dithizone concn. was calculated by reference to a standard curve. This method can be used for the assay of 0·15 μg of dithizone per ml of blood. *In vivo* tests with rabbits showed that dithizone administered in large quantities (40 to 56 mg) disappears from blood so rapidly that 20 min. after its administration only negligible amounts can be detected. K. R. C.

4071. **Determination of the sodium salt of 5-iodosalicylic acid and its metabolites in urine.** P. Formijne and C. F. G. Lehr (Lab. of Gen. Path., Univ. of Amsterdam, Netherlands). *Clin. Chim. Acta*, 1959, **4** (2), 302–306.—*Procedure for free substance*—Acidify the urine (10 ml) with 10 N H_2SO_4 to pH 1, add CCl_4 (20 ml) and shake mechanically for 1 hr. in the dark. Centrifuge, remove the urine layer and shake 7 ml with 7 ml of CCl_4. Dilute the first CCl_4 extract to 10 and determine the u.v. absorption of this (*A*) and of the second CCl_4 extract (*B*) undiluted, at 330 mμ. Evaluate the concn. from a graph covering the range 0 to 10 mg per 100 ml and calculate the concn. in the urine from 20 *A* + *B*. *Procedure for free and conjugated substance*—Boil the urine (10 ml) with 38% HCl (1 ml) for 30 min. Cool, adjust the pH to 1 and continue as described above. Only about 3% of the total substance present was found in the free form.
H. F. W. KIRKPATRICK

4072. **Quantitative determination of Azure A in urine.** B. Klein and M. Weissman (Vet. Admin. Hosp., Bronx, N.Y., U.S.A.). *Clin. Chem.*, 1959, **5** (2), 115–118.—The use of Amberlite XE-96 (or IRC-50) - Azure-A resin in place of quininium exchange resin in the test for gastric acidity without intubation removes the necessity for solvent extraction and fluorimetry. To determine the dye in the urine the following procedure is proposed. To the test and control urines (10 ml) add copper reagent (0·2 g of $CuSO_4.5H_2O$ dissolved in 50 ml of H_2O and made up to 100 ml with conc. HCl (0·1 ml), heat in boiling water for 10 min., cool

e to the original vol. and centrifuge after Dilute 1 ml of each soln. to 10 ml with H_2O read the test against the control at 620 mμ. are a calibration graph by measuring the ction of soln. of dye containing up to 0·05 mg ml of H_2O at 620 mμ against H_2O.

H. W. F. KIRKPATRICK

73. **Paper-disc method for the determination enicillin concentration in whole blood.** M. I. ch-Sherbo, V. I. Batalin and A. F. Bykovskiĭ sk. Med. Inst., USSR). *Lab. Delo*, 1959, (1), 5.—Blood (0·1 ml) is pipetted on to 12-mm le discs consisting of two layers of tough filter- r. After 15 to 20 min. the discs are transferred, neans of a needle, to an agar surface which has seeded with a standard staphylococcus culture. Petri dishes containing the samples are incu- d at 37° for 18 hr.; the diam. of the zones of are then measured and the concn. of penicillin rmined by reference to standard plates con- ng known concn. of the antibiotic. The od is sufficiently sensitive for clinical purposes has an accuracy of ± 3%. K. R. C.

74. **New method for acetone determination in ed air.** D. Glaubitt and J.-G. Rausch- mann (I Med. Univ.-klinik, Hamburg- ndorf, Germany). *Clin. Chim. Acta*, 1959, **4** 65–169.—A constant amount of air (24 litres) pired into a bag fitted with a valve giving little ance to entrance of air. The air is passed a soln. of 2:4-dinitrophenylhydrazine in HCl, the hydrazone formed is extracted with CCl_4. shaking with NaOH the aq. layer is removed the hydrazone is determined by measuring the ction of the CCl_4 soln. at 436 mμ.

H. F. W. KIRKPATRICK

75. **Use of dinitrosalicylic acid reagent for mination of reducing sugar.** G. L. Miller rtermaster Res. and Engng Center, Natick, , U.S.A.). *Anal. Chem.*, 1959, **31** (3), 426– —Potassium sodium tartrate, normally present e reagent, interferes with the protective action e sulphite but is essential for colour stability. difficulty is resolved either by adding it after ormation of the colour or by adding known nts of glucose to the samples to compensate e losses due to its presence.

G. P. COOK

76. **Enzymatic micro-determination of glucose ood and urine.** L. L. Salomon and J. E. on (Dept. of Biochem. and Nutrition, Univ. xas, Galveston, U.S.A.). *Anal. Chem.*, 1959, , 453–456.—Glucose is oxidised with glucose se; the H_2O_2 produced, in the presence of radish peroxidase, oxidises *o*-tolidine to ce a colour which can be measured at 365 mμ mμ.

H. F. W. KIRKPATRICK

77. **Rapid specific method for determination of ccharides in body fluids.** E. Hultman (St. Hosp., Stockholm, Sweden). *Nature*, 1959, 08–109.—The method is based on the con- on of aldoses and ketoses with *o*-toluidine in acid soln. to give a blue-green colour with absorption at 625 mμ. The optimum boiling- for formation of the colour is 8 min. A range of 10 to 400 μg can be read directly in photometer. Other aromatic amines tested similar absorption curves, but were not so ve. The procedure was applied to blood ine samples.

G. P. COOK

4078. **Interference of phosphoric esters of sedoheptulose in the determination of ketohexoses by the method of Roe and Papadopoulos.** A. Bonsignore, S. Pontremoli and E. Grazi (Ist. Chim. Biol., Univ. di Genova, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (11), 509–511.—It is pointed out that, during heptose formation by the non-oxidative route from glucose 6-phosphate, the determination of fructose 6-phosphate by the reaction of Roe and Papadopoulos (*cf. Anal. Abstr.*, 1955, **2**, 430) gives very high values. This is ascribed to the formation of ketoheptose 6-phosphate.

L. ZANONI

4079. **Determination of inulin in blood and urine by means of vanillin in acid medium.** V. E. Levine and W. W. Becker (Dept. of Biol. Chem., Creighton Univ., Omaha, Nebr., U.S.A.). *Clin. Chem.*, 1959, **5** (2), 142–148.—Plasma or urine (1 vol.) is diluted with H_2O (5 vol.) and treated with 10% $ZnSO_4.10H_2O$ soln. (2 vol.) and 0·5 *N* NaOH (2 vol.). The filtrate from the urine is diluted to contain 10 to 100 μg of inulin, that from the plasma is used undiluted. To the filtrate (2 ml) is added 1% aq. vanillin soln. (0·8 ml), and conc. H_2SO_4 (2·2 ml) is then added gradually, shaking after each addition. The mixture is heated in boiling water for 2 min., cooled in ice-water for 2 min. and the extinction is read at 520 mμ, 10 to 20 min. after the removal from the ice-water. A reagent blank and standards are treated similarly.

H. F. W. KIRKPATRICK

4080. **Specific colorimetric method for estimation of phlorrhizin in biological fluids.** F. A. Jenner and D. H. Smyth (Dept. of Physiol., Univ. of Sheffield, England). *Biochem. J.*, 1959, **71** (1), 138–140.—The method is highly specific for phlorrhizin (**I**) and is not given by the aglucone phloretin or any derivative that has been tested. It depends on the formation of a red colour with H_2O_2. The **I** is first extracted into *n*-butanol and then re-extracted with borate buffer, pH 9·1; H_2O_2 is added to the buffer soln. and after 2 hr. at room temp. the extinction is determined spectrophotometrically at 490 mμ, and the amount of **I** ascertained from a standard graph.

J. N. ASHLEY

4081. **Standardisation of the Van den Bergh reaction for serum bilirubin.** R. E. Carter and C. J. McGann (Dept. of Pediat., Univ. of Chicago, Ill., U.S.A.). *Clin. Chem.*, 1959, **5** (2), 106–114.—Calibration curves for the Van den Bergh reaction with commercial preparations of bilirubin showed significant differences from many previously reported. The indicator properties of azobilirubin and the instability of bilirubin in some commonly used solvents are considered to be the cause of these differences.

H. F. W. KIRKPATRICK

4082. **Chromatography of plant pigments.** R. M. Rotfarb. *Byul. Inst. Biol. Akad. Nauk BSSR*, 1956 (1957), (2), 72–74; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (7), Abstr. No. 7865.—An adsorption system is proposed consisting of separate tubes (length 80 mm, diam. 12 to 14 mm) with rubber stoppers. When the adsorbent in one tube is exhausted, another tube with fresh adsorbent is placed under it. The end zones obtained are rejected and the middle zones are re-chromatographed. This method is time-saving and good separations of plant pigments are obtained.

K. R. C.

4083. **Determination of glycerides in blood serum.** L. A. Carlson and L. B. Wadstrom (King Gustaf V Res. Inst., Stockholm, Sweden). *Clin. Chim. Acta*, 1959, **4** (2), 197–205 (in English).—Glycerides are separated from phospholipids by chromatography on silicic acid. After saponification, the glycerol is determined by oxidation with HIO_4 and determination of the formaldehyde formed with chromotropic acid. H. F. W. KIRKPATRICK

4084. **Extraction and titration of poly-unsaturated fatty acids in small amounts of plasma.** S. G. Morris, R. W. Riemenschneider and J. D. Evans (E. Reg. Res. Lab., U.S. Dept. Agric., Philadelphia, U.S.A.). *Arch. Biochem. Biophys.*, 1959, **78** (1), 138–145.—Methods for the extraction of lipids from plasma (1 ml) are investigated and it is concluded that the most satisfactory extraction is attained with dimethoxymethane - methanol (4:1). The extracts are evaporated under N and the residue is dissolved in *iso*octane for measurement of extinction values at five wavelengths. The *iso*octane is then similarly evaporated and the residue isomerised with a KOH reagent (21% in ethanediol) at 180° for 16 min., after which the mixture is diluted with methanol and extinctions at the same five wavelengths are again measured. Poly-unsaturated fatty acids (dienoic to hexaenoic) are calculated from the differences in extinction values by the formulae given. Satisfactory agreement with results obtained by large-scale extraction of fatty acids is attained. W. H. C. SHAW

4085. **Determination of creatinine.** R. Maune (Inst. Ernährung, Potsdam-Rehbrücke, Berlin, Germany). *Ernährungsforschung*, 1959, **4** (1), 90–95.—The red colour produced by creatinine, picric acid, NaOH and water has an absorption max. at 475 to 490 mμ; by statistical calculation the experimental error of the method was reduced by measurement of the colour at max. absorption. Creatinine soln. (10 to 80 mg per 100 ml) (1 ml) is mixed with a cold saturated soln. of picric acid (20 ml) and NaOH soln. (10%) (1 ml). After 20 min. the vol. is made up to 100 ml and after a further 30 min. the extinction is measured at 485 mμ. Results are referred to a standard curve. S.C.I. ABTSR.

4086. **Sensitive spectrophotometric method for adrenaline and noradrenaline.** R. L. Tse and M. J. Oesterling (Dept. of Physiol. Chem., Woman's Coll. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chim. Acta*, 1959, **4** (2), 307–309.—The procedure is applicable to chromatographic separations. Test spots containing 2 to 25 μg of either amine on filter-paper are cut out and placed in 10-ml flasks. To each is added *N* acetate buffer (pH 5·0) (2 ml), the flasks are placed in a water bath at 30° and 0·1 *N* iodine (0·4 ml) is added. After mixing and setting aside for 1 min. for adrenaline (**I**) and 5 min. for noradrenaline (**II**), 0·05 *N* $Na_2S_2O_3$ (0·9 ml) is added and the mixture is shaken until the colour of the iodine is discharged. After setting aside the soln. at room temp. for 18 to 20 hr. the extinction is read against a reagent blank, run concurrently, at 347 mμ (for **I**) or 350 mμ (for **II**).
H. F. W. KIRKPATRICK

4087. **Quantitative determination of adrenalone particularly when associated with adrenaline.** G. Weidner (Pharm. Inst., Humboldt-Univ., Berlin). *Hoppe-Seyl. Z.*, 1958, **312** (4–6), 202–205.—Adrenalone can be reduced polarographically in acetate buffer at pH 4·0 while adrenaline is unaffected. When 10 μg of adrenalone is present as a 1% impurity i adrenaline it can be determined by this means wit an error of ± 3%. Adrenalone in 0·01 *N* HCl show an absorption maximum at 310 mμ which is no shown by adrenaline and enables adrenalone to b determined with an error of ± 2%. F. POWELL

4088. **Determination of choline in blood serum** H. Wachsmuth and L. van Koeckhoven (Hôp Stuivenberg, Anvers, Belgium). *Clin. Chim. Acta* 1959, **4** (2), 206–212.—Serum is deproteinised wit ethanol - diethyl ether and the filtered soln evaporated. The residue is dissolved in satd. aq $Ba(OH)_2$ soln. and saponified for 2 hr. (room temp.) The excess of $Ba(OH)_2$ is pptd. with dil. H_2SO_4 phenolphthalein is added and the soln. is adjuste to just colourless by addition first of *N* NaOH then of *N* HCl. After being centrifuged, the soln is allowed to evaporate spontaneously at a temp ⩾ 40°, the residue is dissolved in ethanol, filtere or centrifuged, and the vol. of the filtrate is reduce to 0·2 ml. Choline is pptd. by slow addition o 10 ml of a soln. consisting of tungstosilicic aci (50 mg) in ethanol (1·5 ml) and diethyl ether (t 10 ml). The ppt. is centrifuged, drained, washe with 2 × 10 ml of ether containing 15% of ethanol and dissolved in warm H_2O (4 ml); 6·7% Na_2S_2O soln. (2 ml) is added and the extinction is read a 650 mμ. An addition of *N* NaOH or pyridine ma be made if required to increase the colour (× 2·6) The method is 600 times as sensitive as th reineckate method. H. F. W. KIRKPATRICK

4089. **High-frequency titration of amino acid and peptides.** F. Oehme (Lab. der Wissenschaftl. Tech. Werkstätten Weilheim/Obb). *Z. Natur forsch.*, 1958, **13b**, 461–462.—Glacial acetic acid i used as the solvent and $HClO_4$ in glacial acetic aci is used as titrant; the end-points are sharp Titration graphs are given for glycine, β-alanin and ϵ-aminocaproic acid. G. P. COOK

4090. **Rapid method for the qualitative analysi of free amino acids in animal tissues by pape chromatography.** J. C. George, S. D. Pishawika and K. S. Scaria (Lab. Animal Physiol., Dep Zool., M.S. Univ. Baroda, India). *Exp. Cell Res* 1958, **15**, 242–244.—Fresh sections of froze muscle, liver and kidney from frog, pigeon and ra were applied directly to circular pieces of Whatma No. 1 filter-paper and chromatograms were ru twice with *n*-butanol - acetic acid - water (4:1:5 The paper was dried at room temp., sprayed wit 0·2% ninhydrin in 95% acetone, and heated f 15 min. at 65°. Comparison was made with alcohol extracts of the same materials, an acid hydrolysa of casein, and a standard mixture of known amir acids; the sections gave all the bands characterist of the extracts. No free amino acid was left in th tissue residue. NUTR. ABSTR. REV.

4091. **Quantitative determination by automat recording of the extinction of amino acids separat by high-voltage electrophoresis.** K. Röwe, Ferber and H. Fischer (Med. Univ. Klinik, Fran furt a. M.). *Hoppe-Seyl. Z.*, 1958, **313**, 174–183. Electrophoresis, at 60 V per cm at pH 2 a 40 V per cm at pH 6, followed by treatment w a copper-containing ninhydrin reagent under rigid controlled conditions, enables the concn. of individual amino acids to be determined. The su of the remaining amino acids not separated these procedures is also obtained. In the procedu described, the extinction values at 310 to 340 n

recorded automatically with a Beckman Spinco lytrol. The whole procedure, including the trophoresis, can be completed in 2 to 3 hr. ear graphs were obtained for 1 to 5 millimoles ach amino acid. Examples are given of the lts obtained with the arginine-rich and the ie-rich fractions of calf thymus histone. It is ested that the technique is capable of further rovement by employing other buffers and using ific colour reactions for various amino acids.
F. Powell

092. **Simultaneous determination of isoniazid D-cycloserine by sodium pentacyanoammino-oate.** V. Scardi and V. Bonavita (Inst. of nan Physiol., Univ. of Naples, Italy). *Clin. n. Acta*, 1959, **4** (2), 161–164 (in English).—vious work (*cf.* Scardi, *Anal. Abstr.*, 1958, **5**, ; Scardi and Bonavita, *Ibid.*, 1958, **5**, 1598) is nded to the determination of D-cycloserine. fresh serum (1 ml) and H_2O (2 ml) in a centri- tube add 10% HPO_3 (1 ml), shake, allow to d for 20 min. and centrifuge. To the super- nt liquid (2 ml) add 16·5% $(NH_4)_2HPO_4$ soln. ml) and 0·4% Na pentacyanoamminoferroate . (*cf.* Scardi, *loc. cit.*) (0·5 ml), shaking after addition. After 10 min. read the extinction 30 mμ (isoniazid) or 630 mμ (cycloserine) and to calibration graphs.
H. F. W. Kirkpatrick

093. **Glutathione, oxidised and reduced, in the n and in isolated cerebral tissue. [Determina- of total and reduced glutathione.]** H. Martin H. McIlwain (Dept. of Biochem., Inst. of hiatry, Maudsley Hosp., London). *Biochem.* 1959, **71** (2), 275–280.—A simple modification ne assay of glutathione by yeast apoglyoxalase scribed. The reduced form of glutathione is coenzyme of glyoxalase, and is a substrate for xalase I, and it is this form which is ordinarily rmined manometrically by the yeast prepara- in the presence of methylglyoxal (**I**). A r relation exists between the CO_2 evolved and amount of glutathione. The oxidised form of thione is reduced by the enzyme preparation they are incubated together for 10 min. in bsence of **I**. The determination of the total ced form is then carried out in the presence of
J. N. Ashley

094. **Protein test indicator.** Eli Lilly & Co. Pat. 814,223; date appl. 20.2.57. U.S.A. appl. 29.2.56.—An indicator for the detection estimation of protein comprises an aq. soln. lining a polychromatic substance which reacts protein to produce a perceptible colour change bromophenol blue or bromocresol green), and fer for maintaining the indicator isohydric at determined pH, *e.g.*, citric acid plus a water- citrate or tartaric acid plus a water-sol. ate.
J. M. Jacobs

095. **Determination of nitrogen in proteins by s of indanetrione hydrate ninhydrin.** S. Jacobs Inst. for Med. Res., Mill Hill, England). *re*, 1959, **183**, 262.—To the sample (0·05 to ml of serum) add H_2SO_4 (1·0 ml) and catalyst O_4 - K_2SO_4 - HgO - Se) (5:15:5:1) (3 to 5 mg), for 45 min., clear with 100-vol. H_2O_2 (0·2 ml) ecessary, and continue heating for 2 hr. ive the digest in 0·4 *M* citrate buffer soln. (0 (50 ml), dilute an aliquot (0·5 ml) to 2·0 ml buffer soln. and add 2% ninhydrin (**I**) soln. (2·0 g of **I**, 50 ml of 2-methoxyethanol, 25 ml of 4 *M* acetate buffer soln. pH 5·5, 25 ml of H_2O and 0·08 g of $SnCl_2.2H_2O$) (2·0 ml). Heat on a water bath at 100° for 30 min., cool and dilute with ethanol - H_2O (1:1, by vol.) (6 ml). Read the colour at 570 mμ. **I** can be purified as previously described (*Analyst*, 1956, **81**, 502); sodium acetate may be recrystallised from ethanol if necessary to reduce the blank colour.
H. F. W. Kirkpatrick

4096. **Micro-determination of ammonium and protein nitrogen.** G. Fels and R. L. Veatch (Vet. Admin. Hosp., Hines, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 451–452.—The method is similar to that of Moore and Stein (*J. Biol. Chem.*, 1948, **176**, 367), the essential difference being the use of a more conc. citrate buffer soln. (0·5 *M* instead of 0·1 *M*). The sample is digested as for the Kjeldahl method, the digest is neutralised with NaOH, and the N determined colorimetrically with ninhydrin.
H. F. W. Kirkpatrick

4097. **Micro-determination of amide nitrogen especially in proteins.** H. Stegemann (Med. Forschungsanst. d. Max-Planck-Gesellschaft, Silikoselabor, Göttingen). *Hoppe-Seyl. Z.*, 1958, **312** (4–6), 255–263.—For the determination of amide-N of amino acids, peptides and proteins, alkaline hydrolysis at $\simeq 20°$ is combined with the diffusion of the NH_3 formed into H_2SO_4. The process is carried out in one flask and is complete in 24 hr. Cystine is decomposed and interferes, but it may be oxidised previously by performic acid. The NH_3 is determined with Nessler reagent. A sample containing from 3 to 30 μg of amide-N is used.
F. Powell

4098. **New method for staining protein electropherograms.** M. N. Egorova (Antibiotics Res. Inst., Min. of Health, Leningrad). *Biokhimiya*, 1959, **24** (1), 127–132.—The use of Acid chrome dark blue (C.I. Mordant Blue 7) as a staining agent is advocated. *Procedure*—Immerse the filter-paper strips for 10 to 15 min. in a 0·08% soln. of the dye in 10% acetic acid. Remove excess of reagent by successive washings (5 to 6) with a 4% soln. of phenol in 10% acetic acid. Remove the protein-fixed colour by extraction with 0·1 *N* NaOH, and measure the extinction of the extract with a green filter (max. absorption at 520 mμ). Alkaline extracts of the reagent are stable for 30 min., but after 60 min. the decrease in extinction becomes significant. The reproducibility of the results, in the determination of the composition of protein fractions in different vol. of blood serum, is good.
K. R. C.

4099. **Zone-sharpening in paper electrophoresis. A method allowing application of dilute protein solutions.** S. Hjertén (Uppsala Univ., Sweden). *Biochim. Biophys. Acta*, 1959, **32** (2), 531–534.—The preliminary concentration of dilute protein soln. is avoided by placing a Cellophane tube (5 cm in circumference), filled with the electrophoresis buffer, under the paper at right angles to the direction of the current a few cm from the line of application of the sample (0·1 to 0·2 ml). Electrophoresis is carried on until the proteins have collected on the line of the tube, which causes zone-sharpening by reduction of the potential gradient where it is in contact with the paper. The Cellophane tube is then removed and normal electrophoresis is followed.
W. H. C. Shaw

4100. **Quantitative determination of tryptophan-rich serum prealbumin on agar.** F. W. Aly and H. Schaupp (Med. Univ.-klinik, Marburg a.d. Lahn, Germany). *Clin. Chim. Acta*, 1959, **4** (1), 88–95.—Electrophoresis on agar is carried out at pH 8·6 for 8 hr. at 90 V (75 mA). The protein is stained with Amido black 10B (C.I. Acid Black 1) and the amount determined planimetrically. In normal persons a range of 12 to 31 mg per 100 ml was found. H. F. W. KIRKPATRICK

4101. **Quantitative method for the separation of thyroid hormones and related compounds from serum and tissues with an anion-exchange resin.** V. A. Galton and R. Pitt-Rivers (Nat. Inst. for Med. Res., Mill Hill, London). *Biochem J.*, 1959, **72** (2), 310–313.—The thyroid hormones and related compounds are adsorbed on Dowex 1 equilibrated with acetic acid - Na acetate buffer of pH 5·6. The iodinated compounds are quant. eluted when the pH is decreased by the use of increasing concn. of acetic acid. Thyroglobulin, and mono- and di-iodotyrosine are eluted at pH 3·6, 3·0 and 2·2, respectively, and thyroxine and tri-iodothyronine at pH 1·4. Iodide remains on the resin. The following advantages are claimed—the iodinated compounds are separated from proteins and lipids; they are eluted with a solvent which can be removed easily, and the residue, after being treated with methanol - conc. aq. NH_3 (3:1), gives a non-viscous extract which can be applied easily to filter-paper for chromatographic analysis. J. N. ASHLEY

4102. **Determination of γ-globulin by zone electrophoresis and by Kunkel's method.** C. A. Payva C. (Inst. Bioquím. y Nutr. y Lab. Cent. Ejército). *Bol. Soc. Quim. Peru*, 1958, **24**, 124–132.—Results obtained by Kunkel's colorimetric method for serum γ-globulin (*Brit. Abstr. C*, 1948, 267) are compared with those obtained by paper electrophoresis in 50 cases of infective hepatitis. Electrophoresis was carried out on Whatman No. 1 paper (35 cm × 4·5 cm) in barbitone buffer of pH 6 and ionic strength 0·1, with the Macheboeff apparatus and 4 mA at 110 V. Zones were located with Naphthalene black 12B (C.I. Acid Black 1) (satd. soln. in methanol containing 10% of acetic acid) applied for 10 min. and then washed with the same solvent. The γ-globulin band was eluted with 10 ml of 0·1 *N* NaOH and measured photometrically at 540 mμ. In the Kunkel test, reagent which had been stored for 1 month in partially filled bottles was used for one group of sera and freshly prepared reagent for a second group. Correlation coeff. were 0·675 and 0·846 for the two groups, and the difference is ascribed mainly to the influence of atmospheric CO_2. E. C. APLING

4103. **Rapid turbidimetric methods for the determination of plasma fibrinogen.** D. A. Podmore (Dept. of Chem. Path., Royal Infirmary, Sheffield, England). *Clin. Chim. Acta*, 1959, **4** (2), 242–245.—Four methods are compared with the Kjeldahl procedure. The results by turbidimetric methods are sufficiently accurate for routine and emergency work. H. F. W. KIRKPATRICK

4104. **Determination of the fibrinogen content of blood plasma by paper electrophoresis.** S. M. Shevchenko (I. M. Sechenov Med. Inst., Moscow). *Lab. Delo*, 1959, (1), 25–27.—Plasma (0·01 ml) was pipetted on to filter-paper strips moistened with a buffer soln. at pH 9·3. Electrophoresis was carried out for 18 hr. at 210 V. The protein was fixed by placing the electropherogram in a desiccato for 30 min. at 105° to 110°. The colour reagen used was Amido black 10B (C.I. Acid Black 1 (200 mg) in ice-cold acetic acid (100 ml) and methanol (900 ml). The electropherograms were developed with a 10% acetic acid - 4% phenol soln Fibrinogen was found to migrate more slowly than γ-globulin, which is contrary to expectation. The strip was sectioned and eluted with 0·1 *N* NaOH An orange filter is recommended for the colou measurement. K. R. C.

4105. **Use of cellulose ion-exchangers for the chromatographic separation of nucleotides.** E. D Korn (Nat. Heart Inst., Bethesda, Md., U.S.A.). *Biochim. Biophys. Acta*, 1959, **32** (2), 554–555.—The separation of adenine, guanine, cytosine and uracil derivatives is attained on cellulose powder treated with 1-chloro-2:3-epoxypropane and triethanolamine and then converted into the formate form. Gradient elution of the compounds is conducted with 0·1 to 0·5 *M* ammonium formate at pH 4·2. The method has the advantage of yielding eluate fractions of small vol. W. H. C. SHAW

4106. **Determination of adenine nucleotides and hexose phosphate esters in tissue extracts.** W. Kunz, W. Schmid and M. Siess (Pharmakol. Inst., Univ. Marburg a.d. Lahn, Germany). *Z. ges. exp. Med.*, 1958, **130**, 328–342 (in German).—The compounds are separated by paper chromatography with acetone - *iso*propyl alcohol - *iso*butyl alcohol - formic acid (30%) (2:1:1:3) as developing solvent The separated compounds are eluted and determined spectrophotometrically and in terms of their P content. NUTR. ABSTR. REV.

4107. **Partial chromatographic separation o pentose- and deoxypentose-nucleic acids.** R. K Main, M. J. Wilkins and L. J. Cole (U.S. Naval Radiol. Defense Lab., San Francisco, Calif., U.S.A.). *Science*, 1959, **129**, 331–332.—Discontinuous elution chromatography with phosphate buffers at pH 6· on calcium phosphate columns separated 73% o tobacco mosaic virus pentosenucleic acid, free fro calf thymus deoxypentosenucleic acid, from mixture of equal parts of these substances. H. F. W. KIRKPATRICK

4108. **Infra-red determination of hydroxy equivalents in steroids.** P. Kabasakalian, E. F Townley and M. D. Yudis (Res. Div., Scherin Corp., Bloomfield, N.J., U.S.A.). *Anal. Chem* 1959, **31** (3), 375–376.—Samples containing 0·1 t 0·4 milli-equiv. of hydroxyl groups are dissolve in pyridine and the extinction is read at the E_{ma} (approx. 3·05 μ) against the solvent. A linea relationship exists between the extinction and th concn. of hydroxyl groups. H. F. W. KIRKPATRICK

4109. **Urinary phenolic steroids. I. Determin tion.** M. F. Jayle, R. Scholler, M. Heron an S. Métay (Lab. d'Hormonol. de l'École de Puéi culture, Fac. de Med., Paris, France). *Clin. Chim Acta*, 1959, **4** (2), 276–296.—Urine is hydrolyse enzymatically with glycuronidase and sulphatas extracted with ether, the ether is washed wi Na_2CO_3 soln. and the oestrogens are extracted wi NaOH. The alkaline soln. is purified by saponific tion or if heavily pigmented by the method Brown (*cf. Anal. Abstr.*, 1955, **2**, 3161). The det mination is made by the Kober reaction, reading 476, 516 and 556 mμ, and applying the All equation. H. F. W. KIRKPATRICK

110. **Quantitative method for the chromatographic separation of 17-oxosteroid sulphates from oxosteroid glucuronides: with observations on behaviour of conjugated corticosteroids on the ne system.** J. J. Barlow and A. E. Kellie urtauld Inst., Middlesex Hosp. Med. Sch., ndon). *Biochem. J.*, 1959, **71** (1), 86–91.—The jugated 17-oxosteroids in an ethanol - diethyl er extract of urine are adsorbed on neutral mina, and the glucuronides are thus separated nt. from the sulphates. The 17-oxosteroid phates are then eluted with 50% ethanol, and glucuronides with *M* acetate buffer, pH 5·0. rapid method is thus provided for the deteration of 17-oxosteroid sulphate: glucuronide os. J. N. Ashley

111. **Colorimetric determination of Δ^4-3-oxoids in the presence of the corresponding dehydro vatives.** M. Bianchi (Vismara Terapeutici, no, Italy). *Chim. e Ind.*, 1959, **41** (1), 33–34.— 2 ml of a soln. containing the steroids in 95% v) ethanol (concn. $\geq$ 100 μg per ml) in a glass-ppered test-tube are added 2 ml of a soln. of -di-*tert.*-butyl-*p*-cresol (0·6%) in ethanol and l of NaOH soln. (20% w/w). The tube is ken and held for 30 min. at 22° to 23°. The 3-oxosteroids give a yellow colour with a maxim at 380 to 385 mμ. L. Zanoni

112. **Changes in oestrogen titre in stored urine.** A. Leon, R. D. Bulbrook and F. C. Greenwood p. Cancer Res. Fund, Royal Coll. of Surgeons, ndon, England). *Nature*, 1959, **183**, 189–190.— increase in oestrogens occurs on prolonged age possibly owing to the microbiological transmation of steroid metabolites. The changes are predictable or constant and analyses should be ed out within 2 days at room temp. or 1 week . H. F. W. Kirkpatrick

113. **Methods of determination of free corticoids in plasma.** J. R. Bierich (Univ. Kinderklin., burg-Eppendorf, Germany). *Endokrinologie*, , **37** (1), 25–35.—Three methods are compared, the Porter - Silber method (*J. Biol. Chem.*, , **185**, 201) and the modifications thereof by r and Busch (*J. Clin. Endocrin.*, 1956, **16**,) and Eik-Nes (*J. Clin. Endocrin.*, 1957, **17**, Although each of the modifications has its ntages the original method remains the most l. Both modifications are long and tedious. For limination of interfering chromogens, the Eik- technique is the best, the benzene - water ation resulting in a cleaner plasma extract. routine clinical determinations the Porter - r method remains the one of choice. B. P. Block

114. **Porter - Silber reaction with C16 substituted eosteroids.** A. Walser and H. P. Schlunke led. Abt., Bürgerspital, Basel, Switzerland). *rientia*, 1959, **15** (2), 71–72.—The determination of 17-hydroxycorticosteroids by the Porter - reaction (*cf. Anal. Abstr.*, 1955, **2**, 142) ads on a typical absorption curve with a max. 0 mμ; 9α-fluoro-16-methylprednisolone gives typical absorption spectrum but not 9α--16-hydroxyprednisolone. It is assumed that es the formation of an osazone a double bond duced between C_{16} and C_{17} which is essential typical Porter - Silber reaction. This has to nsidered when determining the 17-hydroxyosteroids of patients treated with Δ^{16}-corticoids. P. Nicholls

4115. **Determination of cortisone, hydrocortisone, prednisone, prednisolone and 9α-fluorohydrocortisone [fluorocortisone] as 17-oxosteroids after bismuth oxidation.** H. Schreifers and W. Korus (Physiol. Chem. Inst., Bonn). *Hoppe-Seyl. Z.*, 1958, **313**, 66–76.—The bismuthate oxidation technique of Norymberski is adequate for the determination of the total 17-oxogenic steroid content of urine. Other oxidative alterations of the steroid molecule are of little importance in this connection, but are very important if the identification of individual steroids is required. A study was therefore made of the effects of different oxidation times with varying proportions of bismuthate on the steroids listed in the title. Absorption at 220 to 250 mμ, triphenyltetrazolium chloride reduction, micro-Zimmermann reaction and paper chromatography were employed to characterise the derivatives. The absorption curves and R_F values compared with adrenosterone of the 17-oxosteroids derived from the C_{21} steroids used are reported. It was found that oxidation for 1 to 3 min. at a ratio of bismuthate to steroid of 4000 to 1 gave well-defined oxidation products not only with pure substances but also with tissue extracts. F. Powell

4116. **Determination of aldosterone in urine.** T. Di Perri, G. Ravenni and M. Rubegni (Ist. Semeiot. Med., Univ. di Siena, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (7), 351–352.—One-half of a 24-hr. specimen of urine is made up to 1 or 2 litres with satd. NaCl soln. and to pH 1 with conc. HCl, then one-half of this mixture is extracted in a Cohen extractor with $CHCl_3$ for 40 hr. The $CHCl_3$ extract is evaporated *in vacuo* and the residue treated with toluene (2 × 1 ml). The extract is made up to 5 ml with ethanol, and 2 ml chromatographed on paper with 10 μg of a cortisone - cortisol mixture as a control. After 6 hr. the spots are located with a Wood's lamp. The aldosterone has an R_F value intermediate between those of cortisone and cortisol. The spot corresponding to aldosterone is cut out and eluted twice with 1 ml of 95% ethanol for 2 hr. A second chromatographic separation is carried out on this eluate, the spot containing aldosterone being cut out and eluted with abs. ethanol. To 2 ml of eluate are added 0·25 ml of tetramethylammonium hydroxide (1 ml of 10% aq. soln. with 9 ml of 95% ethanol) and 2 ml of 5% ethanolic blue tetrazolium soln. The test-tube is set aside in the dark for 25 min. and 1 ml of acetic acid is then added. The reading is made at 510 mμ against an eluate of the paper alone. L. Zanoni

4117. **Determination of aldosterone in homogenised suprarenal gland.** T. Di Perri, G. Bertelli, G. Ravenni and M. Lodone (Ist. Semeiot. Med., Univ. di Siena, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34**, (7) 1366–1370.—The (rabbit) suprarenal gland is homogenised with 5 ml of normal saline soln. and diluted to 100 ml. This soln. is made up to 500 ml with satd. NaCl soln., and extracted twice with $CHCl_3$ in a Cohen extractor. The $CHCl_3$ soln. is washed first with *N* NaOH (25 ml) and then with satd. NaCl soln. (50 ml), and then evaporated *in vacuo*. The residue is treated with 2 ml of methanol (50% v/v) and extracted twice with 0·5 ml of toluene and light petroleum. An aliquot of the extract (0·1 or 0·2 ml) is chromatographed on Whatman No. 1 paper, with a drop of a cortisone - cortisol mixture on the same paper as a control. The chromatogram is developed with toluene - *n*-octanol - methanol - H_2O (49 : 1 : 35 : 15).

The paper is dried and the spots are located in u.v. light; the aldosterone spot is situated between those of cortisone and cortisol. The aldosterone spot is eluted twice with toluene - light petroleum - methanol - H_2O (7:3:5:5) and determined by the method of Di Perri *et al.* (*Anal. Abstr.*, 1959, **6**, 4116). L. ZANONI

4118. **Enzymic studies of X-irradiated cornea and lens with special reference to glutathione reductase.** [**Assay of reductase.**] R. E. Kuhlman and R. A. Resnik (Ophthalmol. Branch, Nat. Inst. of Neurol. Diseases and Blindness, Bethesda, Md., U.S.A.). *Biochem. J.*, 1959, **72** (2), 261–265.—A sensitive assay for the enzyme in tissues is described. Pre-dialysis is not needed, and inhibition due to reduced glutathione is decreased by working with very dil. soln. Samples of corneal epithelium (1 to 2 μg) or lens homogenate (2 μl containing 50 to 200 μg, wet wt., of lens) are placed in micro test-tubes and covered with an ice-cold medium (10 μl) that contains 0·1 *M* sodium phosphate buffer, pH 7·2, 0·05% of bovine serum albumin, 0·5 m*M* EDTA (disodium salt), 25 m*M* nicotinamide, 1·5 m*M* glutathione and 0·4 m*M* reduced triphosphopyridine nucleotide (**I**). After being incubated at 38° for 1 hr. the tubes are placed in an ice bath and 2 *N* HCl (3 μl) is added to each to stop the reaction. The tubes are then kept at 38° for 3 to 4 min. to destroy any remaining **I**. Aliquots (10 μl) from each tube are added to 10 *N* NaOH (50 μl) in 3-ml fluorimeter tubes and the contents are mixed thoroughly. This converts the oxidised triphosphopyridine nucleotide (**II**) produced in the enzymic reduction of glutathione into a form which can be measured fluorimetrically (Lowry *et al.*, *J. Biol. Chem.*, 1957, **224**, 1047). After 1 hr. at room temp. the samples are diluted with water (1 ml) and are read in a Farrand fluorimeter with primary filter Corning No. 5860 and secondary filters No. 3387, 5562 and 4308. Controls and standards are prepared by adding water (1 μl) and 1 μl of a standard that contains 3 micromillimoles of **II** to the incubation medium containing no sample. J. N. ASHLEY

4119. **Determination of serum lipase with phenyl laurate as substrate.** H. J. Raderecht and H. Moskau (Physiol.-Chem. Inst. d. Humboldt-Univ., Berlin, Germany). *Clin. Chim. Acta*, 1959, **4** (2), 221–226.—The preparation of the substrate is described. Serum (0·2 ml) is incubated for 30 min. at 37° with substrate buffered by acetate to pH 7·4, Na cholate (100 mg) being added to activate lipase. The phenol liberated is determined with Folin's reagent. The enzyme activity is directly proportional to the phenol liberated and to the time of incubation. H. F. W. KIRKPATRICK

4120. **New method for the determination of the activity of proteolytic enzymes.** A. I. Korotyaev and M. L. Khanin (Kubansk. Med. Inst., Krasnodar, USSR). *Lab. Delo*, 1959, (1), 5–7.—This method is based on the fact that the activity of proteolytic enzymes is directly proportional to the number of micro-organisms digested in a given time. *Procedure*—Prepare a bacterial suspension containing 10^{10} bacilli per ml from a fresh 24-hr. agar culture by boiling in physiological saline for 30 min. Add 0·1 *N* HCl (8 ml) and gastric juice (1 ml) to 1 ml of the bacterial suspension and immediately measure the extinction of the suspension in a Pulfrich photometer. Place the suspension in a thermostat at 37° and measure the extinction again after 2 hr. The amount of pepsin in 1 ml of gastric juice i obtained from the formula $E = 100\ (C - C_1)/C$ where E is the amount of pepsin in 1 ml of gastri juice, C is the first extinction and C_1 the second extinction. When this method is used to measur the proteolytic activity of pancreatic juice, 0·5% $NaHCO_3$ soln. is added in place of 0·1 *N* HCl and the time between the measurements reduced to 1 hr. A nephelometer may be used if a photometer is not available. The method may be used for all kinds of proteolytic enzymes. K. R. C.

4121. **Determination of peptic activity.** P. A Nuzhdin and P. D. Rod'kin (Omsk. Vet. Inst. USSR). *Lab. Delo*, 1959, (1), 3–4.—*Procedure*— Dilute 20 ml of blood serum 5 times, add 5% sulphosalicylic acid soln. dropwise to an aliquot until pH 2 is reached and warm to 38° to 40°; then add the enzyme (*e.g.*, 3 ml of gastric juice). After a known time (*e.g.*, 1 hr.) measure 5 ml into each of two titration flasks. Add an excess of neutralised 40% formaldehyde soln. (6 or 7 ml in the case cited) to one titration flask, and titrate the contents of each flask with 0·1 *N* NaOH. The quant increase of the carboxyl groups from the peptic hydrolysis of proteins is obtained from the difference in titres with and without formaldehyde. K. R. C.

4122. **Modified method for determination of uropepsin.** K. E. Arensburger (685 Belden St., Monterey, Calif., U.S.A.). *Clin. Chem.*, 1959, (2), 135–141.—The method of Gray *et al.* (*Neu Eng. J. Med.*, 1954, **251**, 835) is modified by the introduction of a correction for the colour intensity of the urine, termed the chromogenic index (C.I.). The formula used is—$A = R - (R \times \text{C.I.})/10$ A being the corrected result, and R the observed result. This correction reduces error considerably especially in conc. urines. The C.I. is determined by diluting 1 ml of filtered urine with 9 ml of 0·1 *N* HCl and reading the colour at 420 mμ against 0·1 *N* HCl; this reading is evaluated by a standard graph prepared with $K_2Cr_2O_7$ soln. (10 m of 1% aq. soln. and 2 drops of conc. H_2SO_4 diluted to 100 ml with H_2O; 1 ml diluted with 9 ml of water ≡ C.I. of 1). H. F. W. KIRKPATRICK

4123. **Rapid and sensitive spectrophotometric method for the assay of chymotrypsin.** C. Martin, J. Golubow and A. E. Axelrod (Biochem Dept., Univ. of Pittsburgh Sch. of Med., Pa U.S.A.). *J. Biol. Chem.*, 1959, **234** (2), 294–298.— N-Benzyloxycarbonyl-L-tyrosine *p*-nitrophen ester is used as a substrate for the determination chymotrypsin by the spectrophotometric measur ment at 400 mμ of the initial rate of liberation *p*-nitrophenol. The measurement at 275 mμ of th rate of substrate disappearance is an equal effective method of assay. Either method can b used for the determination of $< 4{\cdot}5$ mμg of th enzyme. J. N. ASHLEY

4124. **Determination of alkaline phosphatase blood plasma.** Z. M. Pavlova (Inst. Diet. AM Moscow). *Lab. Delo*, 1959, (1), 37–38.—The activi of plasma phosphatase decreases rapidly wh blood is suspended in physiological saline, especial in the first 10 hr. If a 1:20 dilution of plasma made in chilled physiological saline, which is th centrifuged at 2000 to 3000 r.p.m., the phosphat suffers practically no destruction even after 24 h if the supernatant liquid is kept at a temp. of 2° 4°. K. R. C.

25. **Adaptation of an alkaline phosphatase iod for automatic colorimetric analysis.** W. H. sh, B. Fingerhut and E. Kirsch (Dept. of Path., e Univ. of New York, Brooklyn, U.S.A.). . *Chem.*, 1959, **5** (2), 119–126.—The method l and King (*cf. Anal. Abstr.*, 1955, **2**, 1895) is ɔted to the automatic device of Skeggs (*Amer. J. . Path.*, 1957, **28**, 311).

H. F. W. KIRKPATRICK

126. **Improved method for the determination of inesterase activity in serum.** F. Rappaport, Fischl and N. Pinto (Beilinson Hosp., Petah va, Israel). *Clin. Chim. Acta*, 1959, **4** (2), 221–—The buffer soln. is prepared by mixing a soln. 3·65 g of Na_2HPO_4 and 0·43 g of KH_2PO_4 in ml of H_2O with a soln. of 0·30 g of *m*-nitro-ıol in a similar vol. of H_2O, adjusting the pH to with 0·1 *N* NaOH and diluting to 1 litre. *cedure*—Place 0·9% NaCl soln. (0·1 ml) in each wo test-tubes [for blank (*B*) and test (*T*) soln.]. each add serum (0·1 ml), and inactivate *B* by ing in a water bath at 60° for 3 min. Cool, buffer soln. (2·5 ml) and 15% acetylcholine ride soln. (0·1 ml) to each tube and incubate 5° for 30 min. Read extinctions with a blue r and deduct the blank reading from that of test. *Calibration*—Into each of 11 tubes place er soln. (2·5 ml), inactivated pooled serum ml) and acetylcholine chloride soln. (0·1 ml). he first tube (blank) add H_2O (0·1 ml) and to others acetic acid (0·1 ml) in increasing concn. 0·01 *N* to 0·10 *N*, with increments of 0·01 *N* tube to tube. Each increment represents nits. Read the extinctions as before, deduct the k and plot a curve. Normal values by this ıod are 40 to 80 units.

H. F. W. KIRKPATRICK

127. **Micro-determination of glutamic - acetic transaminase in blood serum.** F. nann, M. H. Brookes and C. A. Storvick gon Agric. Exp. Sta., Corvallis, U.S.A.). *Chem.*, 1959, **5** (1), 57–61.—The method ibed is an adaptation of the method of Karmen *lin. Invest.*, 1955, **34**, 131).

H. F. W. KIRKPATRICK

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e *also Abstracts*—**3893**, Determination of ^{45}Ca ological materials. **3894**, Determination of Ca ological materials. **3898**, Determination of in urine. **3966**, Determination of Mn in able matter. **4181**, Thiamine in biological .. **4211**, Partition chromatography in plant ces. **4219**, Combined paper- and agar-electro-sis apparatus.

Pharmaceutical analysis

128. **Ion exchange and spectrophotometry in idic pharmaceutical analysis.** E. Häberli and éguin (Anal. Lab., Siegfried A.-G., Zofingen, erland). *Pharm. Acta Helv.*, 1959, **34** (2), .—Methods are described for the quant. sis of alkaloids, purines, barbiturates and sic drugs by ion-exchange separation on x 50-X2 and 1-X1 resins, followed by spectro-metry of the eluates. A. R. ROGERS

129. **Bromoplatinic acid as a new reagent for etection of alkaloids and organic bases.** G. (Ist. di Chim. Agrar. dell'Univ., Bologna). *chim. Acta*, 1959, (2), 214–220 (in German).—The sensitivity of bromoplatinic acid (**I**) is greater than that of chloroplatinic acid (**II**). In most cases a 2% soln. of **I** instead of a 10% soln. of **II** is suitable for the detection of strychnine, brucine, quinine and piperazine, and of heterocyclic bases, pyridine, α-picoline, lutidine, collidine, etc.; characteristic orange - red crystals are formed. With **I**, the presence of 2 mg of pyridine per ml can be detected. B. B. BAUMINGER

4130. **Paper-electrophoretic identification of micro quantities of some toxicologically significant alkaloids and comparison with chromatography.** C. Buff, J. Orantes and P. L. Kirk (School of Criminol., Univ. of California, Berkeley, U.S.A.). *Microchem. J.*, 1959, **3** (1), 13–17.—The chromatographic procedure of Goldbaum and Kazyak (*cf. Anal. Abstr.*, 1957, **4**, 240) at pH values of 3·0 and 5·0 (McIlvaine buffers) and 6·5 and 7·5 (Sörensen buffers) has been compared with the electrophoretic technique of Karler *et al.* (*cf. Anal. Abstr.*, 1957, **4**, 1066) with the same buffers. The alkaloids studied were atropine, morphine, emetine, strychnine, cocaine, diamorphine, quinine and yohimbine (50-μg quantities), with codeine as a reference standard. Electrophoresis rates (mm per min.), R_F values and codeine ratios are tabulated for each of these buffers. Satisfactory identification could be obtained by each of the procedures mentioned, but electrophoresis was more reproducible, and therefore more reliable for identification than chromatography, and also much more rapid (1 hr. as compared with 15 hr. for chromatography).

R. E. ESSERY

4131. **Quantitative determination of the morphine content of opium by paper chromatography and paper electrophoresis.** A. B. Svendsen and K. Bergane (Pharm. Inst., Univ., Oslo). *Bull. Narcotics*, 1958, **10** (4), 17–19.—The powdered opium (0·5 g) is ground with water (1 ml) and mixed with acetous Al_2O_3 (Woelm, Eschwege) (10 g). The mixture is percolated with water until 25 ml of filtrate is collected. A drop of this soln. (10 μl) is developed with butanol - acetic acid - water (4:1:5). Morphine forms a spot having an R_F value of 0·45. The spot is developed with 5% aq. $NaNO_2$ soln. giving a yellow colour which changes to red in an atmosphere of NH_3. The spot (and standards prepared in the same way) is cut out and extracted with 1% HCl (2 ml) and 0·5% $NaNO_2$ soln. (2 ml). The soln. is then made alkaline with 5% aq. NH_3 (1 ml) and the extinction measured in a colorimeter with an M465 filter.

N. E.

4132. **Complexometric titration in analysis of organic pharmaceuticals. II. The assay of strychnine salts and their preparations.** Chi-Fon Wu and Ju-Cheng Hsu (Nanking Coll. of Pharm., China). *Acta Pharm. Sinica*, 1959, **7** (2), 78–83.—The proposed method is based on the pptn. of strychnine salts with $HgI_2.2KI$ and subsequent titration of the liberated Hg^{2+} with EDTA. *Procedure*—Pipette 0·0125 *M* $HgI_2.2KI$ (10 ml) into a 50-ml flask, add 0·2% $NaHSO_3$ soln. (5 drops), and shake. Then add the sample soln. (≃ 30 mg of strychnine salt), HCl (1 + 2) (2 ml) and water to the mark. Shake for 1 min. then set aside for 15 min. Filter, discard the first 10 ml and pipette 25 ml of the subsequent filtrate into a 250-ml conical flask. Add 10% KCl soln. (5 ml), 5% $KClO_3$ soln. (6 ml), conc. HCl (3 ml) and water (30 ml). Boil for 1 to 3 min. until the soln. is colourless and clear. Cool to room temp.; add water (70 ml), 20% phenol

soln. (3 drops) and methyl red indicator (2 drops). Neutralise with 10% NaOH soln. and then re-adjust to a pink colour by addition of 2 *N* HCl. Add aq. NH_3 - NH_4Cl buffer soln. (20 ml), 0·1% Eriochrome black T indicator (2 ml) and methyl red indicator (2 ml). Titrate with 0·0025 *M* EDTA (disodium salt) (containing 0·25 g of $MgCl_2.6H_2O$ per litre and adjusted to pH 6·0 to 6·5 with 10% NaOH soln.) under artificial light until the colour changes from olive green through brown, red, brown to green. Carry out a blank test concurrently. The method is simple, rapid and accurate. The results of recovery tests in 2 samples of commercial strychnine salts by the proposed method agree within ± 0·2% with those by three established methods. S. H. Yuen

4133. **New method for the determination of reserpine in pharmaceutical preparations.** V. Scarselli (Lab. Ric. Ist. Giuliani, Milano, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (18), 1132–1134.—To the sample, containing from 200 to 800 μg of reserpine, in a glass-stoppered test-tube, are added 5 ml of acetic anhydride and 5 ml of a 2% (v/v) soln. of amyl nitrite in $CHCl_3$. After being shaken, the tube is placed in a water bath at 40° for 90 min.; it is then cooled and the extinction is read at 475 mμ. The error is up to ≃ ± 0·9%. In the presence of interfering substances (*e.g.*, phenazone and other pyrazolone derivatives), a preliminary extraction with $CHCl_3$ must be made. L. Zanoni

4134. **Rapid and simple colorimetric determination of reserpine in pharmaceutical preparations.** A. W. M. Indemans, I. M. Jakovljevic and J. J. A. M. van der Langerijt (Lab. der Nederlandse Apothekers, Utrecht, Holland). *Pharm. Weekbl.*, 1959, **94** (1), 1–8.—Prepare an extract of the sample (containing 50 to 300 μg of reserpine) in glacial acetic acid (6 ml), add 2% aq. $NaNO_2$ soln. (0·4 ml) and heat in a bath of boiling water for 5 min. Cool to room temp., dilute with H_2O to 40 ml, extract with $CHCl_3$ (10 ml) and measure the extinction of the $CHCl_3$ layer at 465 mμ. The orange colour produced by the hydrolytic degradation products is not extracted by $CHCl_3$. Interference by primary oxidation products of reserpine is corrected by measurement of the extinction at 390 mμ of a $CHCl_3$ extract of a soln. of the sample in dil. acetic acid. A. R. Rogers

4135. **Colorimetric method for the determination of the activity of cardiac drugs.** V. N. Kobalenko. *Aktual'n. Vopr. Perelivaniya Krovi*, 1958, (6), 312–324; *Ref. Zhur., Khim., Biol. Khim.*, 1959, (8), Abstr. No. 9298.—The method is based on the colour reaction of glycosides with picric acid. Glycosides are determined in preparations of strophanthus and adonis and the extinctions are measured at 490 to 550 mμ. Calibration curves for soln. of strophanthin-K and cymarin are plotted. The results obtained are more accurate than those obtained by biological methods. K. R. C.

4136. **Unit cell dimensions and X-ray powder data of prednisolone acetate.** R. A. Pasternak (Stanford Res. Inst., Menlo Park, Calif.). *Anal. Chem.*, 1959, **31** (5), 959.—The results of an X-ray crystallographic study are reported.

4137. **New quantitative analytical method for santonin in *Artemisia incana* Keller.** Feng-Yung Fu, Min-Ju Yen and Fung-Mei Wang (Dept. of Med. Plants, Chinese Acad. of Med. Sci., Peking *Acta Pharm. Sinica*, 1959, **7** (2), 53–58.—Th sample (0·25 g) is extracted with boiling Ca(OH soln. The soln. is filtered and Ca santonate in th filtrate is adsorbed on animal charcoal (1 g). Th adsorbed santonate is treated with 1% HC the free santonin (**I**) is extracted with $CHCl_3$, and th $CHCl_3$ soln. is evaporated to dryness. **I** i the residue is treated with dil. H_2SO_4 (1 + 1) (3 m and 0·8% $FeCl_3$ soln. (0·1 ml) and determine colorimetrically. The method has been applie to 4 samples of *Artemisia incana* Keller with result agreeing well with those obtained by an establishe gravimetric method. The coefficient of variatio is ± 4·9%. S. H. Yuen

4138. **Polarographic determination of noradren aline, adrenaline and Aludrine [isoprenaline].** I Ramonțian, A. Gross and I. Schwartz. *Rev. Chim. Bucharest*, 1959, **10** (2), 111–115.—These sub stances show two polarographic steps, proportiona to their concn., by means of which the ketones ca be determined in admixture with the correspondin alcohols. *Procedure*—The soln. of adrenalin sulphate, isoprenaline sulphate or noradrenalin hydrochloride (2 ml) is mixed with basal soln (2 ml); 3 drops of 1% gelatin soln. are added t suppress the maxima, the soln. is de-aerated b bubbling with H for 5 min., and the polarogram recorded in a Heyrovský apparatus, type V301, a 4 V, with a galvanometer of sensitivity of 1/200 and a mercury flow of 1 drop per 3 sec. Figure are recorded for $E_{\frac{1}{2}}$ *vs.* the N.C.E., with 8 basa soln. at pH from 1·5 to 9·5. H. Sher

4139. **High-frequency titrations for the determina tion of basic antibiotics.** F. Oehme (Lab. de Wissenschaft.-Tech. Werkstätten, Weilheim/Obb *Z. Naturforsch.*, 1958, **13b**, 462.—Glacial aceti acid is used as the solvent in the high-frequenc titration of basic antibiotics such as oxytetracyclin and actinomycin; $HClO_4$ in glacial acetic acid used as the titrant. End - points are sharp, an can be visually observed by the deflection of th ammeter. Titration diagrams are given. G. P. Cook

4140. **Spectrophotometric determination of d hydrodeoxystreptomycin.** Hatsuko Ikeda an Hiroshi Ikeda. *J. Sci. Res. Inst., Tokyo*, 1957, **5** 234–241.—The acid-hydrolysed product of d hydrodeoxystreptomycin (**I**) has a characteristi u.v. absorption with small changes in extinctio within a limited concn. range. Samples (13 15 mg) are treated with 2 ml of 0·5 *N* H_2SO_4 a heated for 2 hr. at 100°. After cooling, the co tents are diluted with water to 50 ml and t extinction is measured at 265 mμ against a blan A standard curve is obtained by similarly treati aliquots of a standard **I** sulphate soln. Chem. Abstr.

4141. **High-frequency titrimetric determinati of ferric ions and its application to the determinati of oxytetracycline.** K. Hochmann and I. Bay (Lab. of the Pharmacopœia Commission, Budape Hungary). *Z. anal. Chem.*, 1959, **166** (2), 88–92. Ferric ions (0·01 *M* soln.) are accurately determi at pH 2 by high-frequency titration with EDTA Pungor's apparatus (Pungor and Huber, *Ib* 1957, **154**, 1); the same technique can be appl to Fe^{3+} complexes, *e.g.*, to Fe^{3+} - oxytetracycli and thus to the determination of oxytetracycl (accuracy to within ± 0·5%). *Procedure*—Disso

tetracycline (0·1 g) in 0·1 *N* HCl (10 ml) and te to 25 ml. Neutralise an aliquot (5 ml) with N NaOH to bromophenol blue, add 0·01 *M* I_3 (10 ml), dilute to about 120 ml and titrate r 10 min. with 0·01 *M* EDTA. Correct the lt by a blank determination without oxytetra· ine. J. P. STERN

142. **Spectrophotometric determination of fungi- n [nystatin] in culture fluid and comparison i the biological method.** D. Doskocilova and Hess (Antibiotics Res. Inst., Prague). *Anti- tiki*, 1959, **4** (1), 50–54.—*Procedure*—Culture l (2 ml) is mixed with anhyd. methanol (8 ml), rously shaken for 10 min. and centrifuged. mycelium is extracted again with 80% methanol ml). The extracts are combined and made up 25 ml with 80% methanol. An aliquot of this . (5 ml) is made up to 50 ml with anhyd. hanol and the extinction measured at 304·5, and 318 mμ. When the ratio of the extinction 312 mμ to that at 304·5 mμ lies between 0·75 0·85, the nystatin concn. of the culture fluid nits per ml is given by 125 $(E_{304·5} - E_{312})/0·0106$. *plified procedure*—Culture fluid (2 ml) is mixed h anhyd. methanol (8 ml), shaken for 10 min. centrifuged. An aliquot (2 ml) of the soln. is le up to 50 ml with H_2O and the extinction asured at 306, 314 and 321 mμ. The formula hen $125(E_{306} - E_{314})/0·0075$. The error is ≃ . The first method was compared with a ogical assay in which plate diffusion tests and train of *Candida albicans* resistant to cyclo- imide were used. Both methods gave com- able results throughout the fermentation, but spectrophotometric method had the advantage peed and simplicity. K. R. C.

143. **New procedure for the determination of biturates in non-aqueous medium.** J.-A. Gautier, Pellerin and J. Pineau (Fac. de Pharm., Paris). *. Pharm. Franç.*, 1958, **16** (11), 625–630.— barbituric acids react with $AgNO_3$ in pyridine . to liberate two H^+ per mol., and the mono- um salts react to liberate one. The free H^+ titrated with ethanolic NaOH soln. *Pro- re*—The barbituric acid (≃ 1 milli-equiv.) is olved in 5 ml of pyridine and 10 ml of 0·5 *N* $AgNO_3$ is added. The soln. is titrated with 0·1 *N* nolic NaOH, either potentiometrically, or with 5 drops of 1% ethanolic thymol blue as cator. If free acid and sodium salt are present ther, the sodium salt can be determined by tion in anhyd. acetic acid (Gautier and Pellerin, . *Pharm. Franç.*, 1952, **10**, 401). In simple arations the barbiturates are extracted with line and titrated as before, and in more complex ures the pyridine soln. is neutralised with H soln. to thymol blue before adding the $AgNO_3$ soln. This converts free barbituric acids the monosodium salts. Results with a number ure barbiturates are given showing errors of 1%. E. J. H. BIRCH

144. **Preliminary assay for the separation of turates by means of electrophoresis.** A. Calo', Mariani and O. M. Marelli (Ist. Sup. Sanità, a, Italy). *R.C. Ist. Sup. Sanit.*, 1957, **20**, 315.—Electrophoresis is used for the separa- of barbiturates of pharmaceutical interest. results are obtained by working at pH 9 with Britton and Robinson buffer soln. (*cf. J. Soc.*, 1931, 1456) on S. & S. 2045b paper at V per cm for 12 to 14 hr. It appears possible to separate only barbiturates containing *cyclo*hexenyl and *cyclo*heptenyl groups from those with other substituting groups. L. ZANONI

4145. **Differentiation of barbiturates for clinical and medico-legal purposes.** G. L. Plaa, F. B. Hall and C. H. Hine (Univ. California Med. School, San Francisco). *J. Forensic Sci.*, 1958, **3**, 201–209.—Barbituric acid derivatives are isolated from biological material by extracting a tungstic acid filtrate with diethyl ether, and purifying the residue by ascending paper chromatography (*cf.* Algeri and Walker, *Amer. J. Clin. Path.*, 1952, **22**, 37). The spot is made visible by using a "Mineralite" source whose primary absorption is at 2550 A. The spot is then extracted into 0·45 *N* NaOH and its u.v. absorption characteristics are determined by using Goldbaum's technique of absorption ratios (*cf. Anal. Chem.*, 1952, **24**, 1604). The barbiturate can then be characterised. The described technique permits a qual. and quant. determination of barbiturates in biological material with only one sample. CHEM. ABSTR.

4146. **Micro-reaction for methylphenobarbitone.** H. M. Romijn. *Pharm. Weekbl.*, 1959, **94** (4), 102–103.—When a mixture of methylphenobarbitone with Co acetate is warmed with a drop of 10% aq. NH_3, a red - purple colour (Parri's reaction) is produced within 1 min., and, on cooling and scratching the tube, characteristic parallelogram crystals with rounded obtuse angles are formed. The reaction can be carried out with 1 to 2 mg if the concn. is high. Characteristic crystals are also given by other barbiturates. P. RENTENAAR

4147. **Photo-electric colorimetric method for the estimation of procaine.** M. Z. Barakat, S. K. Shehab and M. El-Shabrawi (Fac. of Vet. Med., Giza, Cairo, Egypt). *Drug Standards*, 1959, **27** (1), 27–30.—To determine procaine hydrochloride (**I**) in injections, mix the sample (5 ml, containing ≃ 0·5 mg of **I**) with a 0·2% aq. soln. of Na 1:2-naphthaquinone-4-sulphonate and measure the transmission with a blue - green filter (Lumetron 490). Calculate by reference to a calibration curve. To determine procaine in ointments, dissolve the sample (1 g) in diethyl ether or ethyl acetate (150 ml), extract with H_2O or 0·5% aq. HCl (3 × 30 ml), and use an aliquot of the combined aq. phases. A. R. ROGERS

4148. **Non-aqueous titration of some 5-amino-acridine derivatives.** L. A. Phoryles and N. Cohen (Kupath-Holim Sick Fund, Supply Dept., Tel Aviv, Israel). *Drug Standards*, 1959, **27** (3), 92–93.—Mepacrine hydrochloride (**I**) and Rivanol (2:5-diamino-7-ethoxyacridine lactate) (**II**) have been successfully titrated with 0·1 *N* $HClO_4$ in glacial acetic acid to crystal violet indicator. Two of the three NH_2 groups in **I** and one of the two in **II** react. The results for **I** (5 samples) vary from 100·3 to 100·7%. N. E.

4149. **Determination of sulphonamides that have a hypoglycæmic effect.** R. Simionovici and I. Conu. *Rev. Chim., Bucharest*, 1959, **10** (2), 107–108.—Two methods are given for sulphonamides of the type N-butyl-N′-toluene-*p*-sulphonylurea (tolbutamide) and N-*cyclo*hexyl-N′-toluene-*p*-sulphonylurea. *Direct titration procedure*—The substance (0·3 g) is dissolved in 25 ml of acetone previously neutralised to cresol red, and titrated

with 0·1 *N* NaOH to a rose - violet colour. *Procedure for the method of hydrolysis*—The substance (0·3 g) is treated with 10 ml of ethanediol and 2 ml of conc. HCl, and heated in an oil bath at 120° to 122° for 30 min. The mixture is then diluted with water, treated in a Kjeldahl flask with 15 g of NaOH, and the amine is distilled off into 25 ml of HCl (0·1 *N*), the excess of which is determined by titration. Results obtained on these substances show max. errors of ± 1·0%. H. Sher

4150. **Separation of some phenothiazine derivatives.** A. Calo', A. Mariani and O. M. Marelli (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1957, **20**, 987–992.—The characteristics of promazine (**I**) are compared with those of chlorpromazine (**II**). The behaviour of **I** is similar to that of other phenothiazine derivatives not containing chlorine in the molecule. As compared with **I**, **II** exhibits a greater electrophoretic mobility and a more intense and sensitive colour reaction on oxidation with HIO_3. L. Zanoni

4151. **Polarographic oxidation of phenothiazine tranquillisers.** P. Kabasakalian and J. McGlotten (Res. Div., Schering Corp., Bloomfield, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 431–433.—Polarography is carried out at a rotating gold-wire electrode in aq. H_2SO_4 soln. Anodic waves of 14 substitution compounds of phenothiazine are described and shown to be suitable for analysis. H. F. W. Kirkpatrick

4152. **Electrophoretic separation of chlorpromazine and its colorimetric determination.** A. Calo', A. Mariani and O. M. Marelli (Lab. Chim., Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1957, **20**, 802–810.—*Procedure*—To 1 ml of aq. chlorpromazine hydrochloride soln. are added 2·5 ml of H_3PO_4 (d = 1·7) and 1 ml of HIO_3 (10%); a red colour with an absorption maximum at 526 mμ is developed. By this method it is possible to determine chlorpromazine in the presence of barbiturates and rauwolfia alkaloids. In the presence of compounds having a similar chemical constitution (*e.g.*, promethazine), electrophoresis on S. & S. paper 2083b in acetate buffer (pH 4) of ionic strength 0·08 is used to separate the chlorpromazine. L. Zanoni

4153. **Detection of meprobamate on paper chromatograms.** H. S. Bedson and T. H. Farmer (London Hosp., London). *Nature*, 1959, **183**, 43.—Spray the dry chromatogram first with 1% NaOCl soln., then with ethanol and finally with 1% starch soln. - 0·05 *M* KI (1 : 1). A. R. Rogers

4154. **New volumetric method for the determination of piperazine adipate.** R. Simionovici, J. Răianu and V. Cuculescu. *Rev. Chim., Bucharest*, 1959, **10** (2), 105–106.—The method is based on the Sörensen 'formol' method for amino acids. *Procedure*—Approx. 0·20 g of the sample is dissolved in 10 ml of water and treated with 10 ml of formaldehyde soln. (35%) previously neutralised to phenolphthalein; 4 drops of an ethanolic soln. of thymolphthalein are added, and the mixture is titrated with NaOH soln. (0·1 *N*) to a standard colour [25 ml of water, 10 ml of neutralised formaldehyde soln., 4 drops of thymolphthalein soln. and 0·2 ml of NaOH soln. (0·1 *N*)]. The standard error is ± 0·31%. H. Sher

4155. **Colorimetric assay for the determinatio of diethylstilboestrol [stilboestrol].** J. D. Duer and B. A. Pappas (Chas. Pfizer & Co. Inc., Brooklyn N.Y.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **4** (1), 13–15.—Stilboestrol (**I**) reacts with H_2SO_4 $FeCl_3$ reagent [add conc. H_2SO_4 (40 ml) to a 10% soln. of $FeCl_3.6H_2O$ (1 ml)] to give a violet colou which obeys Beer's law at concn. in the range 1 t 2 mg of **I** per 25 ml of soln. The method is applie to the assay of implant pellets. The results com pare favourably with those by the methods of th U.S.P. and of Goodyear *et al.* (*cf. Anal. Abstr.* 1955, **2**, 457). *Procedure*—Add the reagent (4 ml in portions with swirling to an ethanolic soln. o the sample (5 ml containing approx. 1·5 mg of **I** and set aside at 20° for 5 min. Dilute with ethano to 25 ml and after a further 10 min. measure th extinction at 558 mμ against a reagent blank. Tak the mean of 3 determinations. A. R. Rogers

4156. **Colorimetric determination of menadion [menaphthone].** F. J. Bandelin and R. E. Pankrat (Control Lab., Flint, Eaton & Co. Inc., Decatur Ill., U.S.A.). *Drug Standards*, 1959, **27** (2), 36–39.— Heat a soln. of menaphthone (≃ 1 mg) in ethano (1 ml) with a 1% soln. of $ZnCl_2$ in conc. HCl (4 ml on a bath of boiling water for 30 min., cool, dilute to 50 ml with ethanol and measure the extinctio at 510 mμ against a reagent blank. The metho has been applied to the assay of oily soln. and tablets. Vitamins K_1 and K_2 give no colour Results are accurate to within ± 1%. A. R. Rogers

4157. **Determination of halogens in iodochlorhydroxyquin [5-chloro-8-hydroxy-7-iodoquinoline].** S. C. Slack and W. J. Mader (Res. Dept., Ciba Pharmaceutical Products Inc., Summit, N.J., U.S.A.). *Drug Standards*, 1959, **27** (2), 33–35.— Slowly add 20% fuming H_2SO_4 (5 ml) to a mixture of the sample (0·3 g) with $CuSO_4$ (0·3 g), starch (0·1 g) and $K_2S_2O_8$ (3 g) and heat under reflux fo 20 min. Cool, add H_2SO_4 (5 ml) and heat until the soln. is clear and light green in colour. Cool, add 20% H_2O_2 soln. (5 ml) and distil into a soln. o As_2O_3 (0·2 g) in 10% NaOH soln. (20 ml). Cool add saturated hydrazine sulphate soln. (5 ml) and distil for 5 min. Cool, add 20% H_2O_2 soln. (5 ml and distil for 5 min. Add conc. aq. NH_3 (1 ml) t the combined distillates, and titrate the iodid amperometrically (rotating platinum electrode with 0·1 *N* $AgNO_3$, with a mercury - mercuri iodide reference electrode. Add conc. HNO_3 (1 ml and 1% aq. gelatin soln. (10 ml) and titrate th chloride in the same way with a S.C.E. as referenc electrode. The results of four assays of one sampl show a coeff. of variation of 0·1% for iodine an 1·1% for Cl. A. R. Rogers

4158. **Simple and rapid method of chemic assay of 5-chloro-8-hydroxy-7-iodoquinolin [iodochlorhydroxyquin]—an amoebicidal drug.** S. Mukerje and A. P. Dey [Anal. Sect., Albert Davi (Laboratories) Ltd., Calcutta]. *Drug Standard* 1959, **27** (1), 18–20.—Fuse the sample (0·5 g or a equiv. amount of powdered tablets) with Na_2C by the B.P. method for the assay of di-iodohydrox quinoline. Cool, extract the residue with H_2 filter and dilute to 250 ml. To determine tot halogen, mix a 100-ml aliquot of the filtrate wit 0·1 *N* $AgNO_3$ (25 ml), acidify with conc. HNO_3 a titrate with 0·1 *N* KSCN. To determine iodi only, neutralise a 100-ml aliquot with HCl, co add conc. HCl (15 ml) and 10% KCN soln. (6 m

titrate with 0·05 *M* KIO_3. The complete assay s 2·5 to 3 hr. and the results agree with those ined by the methods of the U.S.P. and "New Non-official Remedies," 1941. A. R. Rogers

159. **Colorimetric micro-determination of cant substances [1-chloro-1-oximinoacetone dichloroformaldehyde oxime].** P. Malatesta A. Lorenzini (Inst. Pharm. Toxicol. Chem., v. Rome). *Ann. Chim., Roma*, 1958, **48** (12), 3–1341.—1-Chloro-1-oximinoacetone is deter- ed by means of the hydroxylamine formed ether with pyruvic acid) on hydrolysis with . The soln. containing the hydroxylamine is lised to nitrous acid with iodine, and the NO_2^- determined with sulphanilic acid and 1- hthylamine. The extinction is read at 520 mμ. urate results are obtained over the concn. ge of 0·75 to 7·5 μg per ml. Dichloroformalde- e oxime is determined by reaction with 4-(*p*- obenzyl)piperidine and $Hg(CN)_2$ and reading of extinction at 500 mμ. The method is suitable a concn. range of 0·4 to 5·5 μg per ml.
A. G. Cooper

160. **Analysis of dermatologic and cosmetic ment preparations. VI. Ultra-violet absorption ointment bases.** R. Springer and H. Weigand st. Pharm. u. Lebensmittelchem. Univ., achen). *Arch. Pharm., Berlin*, 1958, **291** (11–12), –595.—Wavelengths of max. and min. absorp- of various ointment bases dissolved in $CHCl_3$ given. The values may be used for identifica- purposes. M. H. Sawistowska

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ee also Abstracts—**3898**, Hg in disinfectants. **7**, Determination of methyl salicylate. **4069**, ermination of barbiturates. **4071**, Determination Na 5-iodosalicylate. **4073**, Penicillin in blood. **3**, Determination of adrenaline and noradrena- **4092**, Determination of isoniazid. **4114**, Deter- ation of prednisolone deriv. **4115**, Determina- of corticosteroids. **4120**, Activity of proteolytic rmes. **4176**, Fat-soluble vitamins in aq. dis- ions. **4179**, Assay of thiamine.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

61. Rapid method for the determination of fat odstuffs and industrial products. I. Invernizzi C. Sampietro (Lab. Chim. Prov., Como, Italy). *Lab. Chim. Provinciali*, 1959, **10** (1), 27–32.— *edure*—The sample (5 g) is weighed in a centri- tube and dissolved in 10 ml of warm water. ene (2 × 15 ml) is added to the cooled soln., a pipette, then the tube is stoppered, shaken centrifuged. With the same pipette 15 ml of benzene layer is removed and evaporated at temp., and the residue is dried at 100° and ned. L. Zanoni

32. Application of gas chromatography in the ectionery industry. N. A. Lund (Mars Ltd., h, Bucks., England). *Perfum. Essent. Oil* 1959, **50** (2), 147–152.—Gas chromatography upplement but not entirely replace olfactory ation of essential oils and flavours used in tuffs. Chromatograms of peppermint oils and butter are used to illustrate adulteration. constitution of commercial "citral 99/100%" lf distilled lime oil is investigated and discussed. H. B. Heath

4163. **Composition of honey. IV. Identification of disaccharides.** J. W. White and N. Hoban (E. Reg. Res. Lab., U.S. Dept. Agric., Philadelphia, U.S.A.). *Arch. Biochem. Biophys.*, 1959, **80** (2), 386–392.—By preliminary column chromatography on charcoal followed by chromatography on Whatman No. 1 or 3MM paper and elution with *n*-propanol - ethyl acetate - water (7:1:2) or zone electrophoresis, fractions are isolated and identified by their i.r. spectra and those of their β-octa-acetates. Isomaltose, maltulose, turanose, nigerose, maltose and sucrose were identified in the sample examined. W. H. C. Shaw

4164. **Determination of starch.** R. G. Eeckhaut. *Bull. Éc. Meun. Belg.*, 1958, **20** (3), 39–46 (in French).—To evaluate the various methods of determination, a sample of potato starch was used in which the starch had been determined by difference. Colorimetric methods were not examined but the polarimetric methods of Lintner-Belschner, Ewers and Mannich-Lenz, the gravimetric methods of Mayerhofer, Rask and Baumert-Bode, and acid hydrolysis were compared. The Lintner-Belschner, Ewers, Rask and Baumert-Bode methods did not give satisfactory results. The three methods of choice are the Mannich-Lenz, the Mayerhofer and the acid hydrolysis of Peat and Whelan followed by the determination of glucose by Luff-Schoorl's method. A method is suggested for the determination of starch in the presence of sugars.
Brit. Baking Ind. Res. Ass. Abstr.

4165. **Comparison of the Orange G dye and Kjeldahl methods for determining milk protein.** J. M. Treece, L. O. Gilmore and N. S. Fechheimer (Ohio Agric. Exp. Sta., Wooster, U.S.A.). *J. Dairy Sci.*, 1959, **42** (2), 367–368.—The correlation coefficient of results by the two methods was 0·98. The method in which Orange G (C.I. Acid Orange 10) is used (*cf.* Udy, *Anal. Abstr.*, 1957, **4**, 4118) is preferred for simplicity, rapidity and lower cost.
W. H. C. Shaw

4166. **Spectrophotometric determination of benzoic acid in margarine.** J. B. Roos and A. Versnel (Rijkszuivelstation, Leiden, Holland). *Chem. Weekbl.*, 1959, **55** (7), 67–70.—*Procedure*—Melt the sample (5 g) with 30 ml of methanol, transfer with 30 ml of methanol to a glass-stoppered 100-ml flask, add to the mixture water (5 ml) and 4 *N* H_2SO_4 (0·5 ml), and shake for 1 min. Cool to 20° and make up to vol. with acidified methanol. Cool to solidify the fat, then filter, rejecting the first 20 ml. Measure the extinction of the filtrate in a 1-cm cell at 272 mμ, and convert this value to benzoic acid (g per litre) by a multiplication factor of 5·697. Determine the blank value on an aq. mixture of acidified methanol of the final composition attained in the determination. The maximum analytical error is ⩾ 0·002%. P. S. Arup

4167. **Analytical methods for controlling the purity of erythrosine in the residual baths from the dyeing of cherries.** C. Bersano (Azienda Colori Beghè, Milano, Italy). *Ind. Cons.*, 1959, **34** (1), 11–13.—Several relatively simple analytical methods for controlling the purity of erythrosine (**I**) (C.I. Acid Red 51) are given. For the determination of **I** in residual baths from dyeing cherries, 50 ml of filtered soln. is diluted with boiling water, 10 g of Na tartrate is added, and the soln. is titrated hot, in an atmosphere of CO_2, with 0·1 *N* $TiCl_3$.
L. Zanoni

4168. **Determination of foreign fats in cacao products. IX. Identification by column chromatography of small additions of an English cacao-butter substitute EKE derived from palm oil in the acetone-soluble glyceride fraction obtained from cacao butter at −25°.** A. Purr (Inst. für Lebensmitteltechnol. u. Verpackung, Munich). *Fette, Seif., Anstrichmitt.*, 1959, **61** (2), 119–126.—Low-temp. (− 25°) crystallisation of a 10% soln. in acetone is applied to pure cacao butter, pure EKE (which has a glyceride composition similar to that of cacao butter) and adulterated cacao butter to give glyceride fractions which are then saponified. The fatty acid mixtures are then chromatographed (without removal of unsaponified material) on kieselguhr treated with dichlorodimethylsilane to produce inverse phase conditions. Liquid paraffin is used as the stationary phase and acetone - water mixtures saturated with liquid paraffin as the mobile phase. The separated acids are titrated and identified by mol. wt. determination. Full experimental details are given, together with the results obtained. Chromatograms for the acid mixtures obtained from the three products are shown. The analysis of synthetic fatty acid mixtures confirms the validity of the method.

S. M. Marsh

4169. **The quantitative determination of β-carotene in fruit juices.** A. Daghetta and O. Bruss (Staz. Sper. del Freddo, Milano, Italy). *Ann. Sper. Agr.*, 1957, **11** (3) (Suppl. CXVIII–CXX).—*Procedure*—The sample (50 g) is diluted with 150 to 200 ml of H_2O. The suspension is centrifuged and the residue, after removal of the supernatant liquid, is filtered on a Buchner funnel, then the residue, mixed with anhyd. Na_2SO_4 and ground quartz, is extracted in a Soxhlet apparatus with 100 ml of acetone - light petroleum (3:7) for 1 hr. The solvent is evaporated to remove acetone, and the soln. is made up to 50 ml with light petroleum; 5 or 10 ml of this soln. is chromatographed on a column (10 cm) packed with Al_2O_3 - Celite (1:1), and eluted with light petroleum containing 5% of acetone. The eluate is made up to a known volume and the extinction measured at 450 mμ.

L. Zanoni

4170. **Determination of total and volatile acids in fruit juices and wines.** K. Woidich, L. Schmid, H. Gnauer and H. Woidich (II Chem. Univ.-inst. u. Lebensmittelversuchsanst., Wien, Austria). *Z. LebensmittUntersuch.*, 1958, **108** (4), 363–365.—A titration end-point of pH 7·8 is suggested for wines and the juices of grape, apple, pear, cherry and tomato; and pH 8·4 for the juices of citrus fruit, berries (currants, raspberry, strawberry) and pineapple. Abnormal results are obtained with the juices of barberry and rhubarb, which contain oxalic acid. For the determination of volatile acids in wines, use was made of an apparatus (Woidich, *Dtsch. LebensmittRdsch.*, 1956, **52**, 267) for steam-distilling small quanties of liquids (*e.g.*, 10 ml of wine or 5 ml of an acetified wine) collecting ≃ 120 ml of distillate and, after heating nearly to boiling for a short time, titrating the distillate electrometrically with 0·1 *N* NaOH to pH 8·4.

S.C.I. Abstr.

4171. **The thiobarbituric acid value as a measure of the degree of oxidation of edible fats.** H. Schmidt (Bundesforschungsanst. f. Lebensmittelfrischhaltung, Karlsruhe, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (2), 127–133.—The thiobarbituric acid and phloroglucinol tests are discussed with reference to malonic dialdehyde (**I**) and its tautomers, pyrimidine derivatives and unsaturated fatty acids. It is concluded that both tests are specific for **I** and its tautomers, and can be used for the identification of poly-unsaturated fatty acids in natural and hydrogenated fats and pure oleic acid. Since these acids give rise to **I** during the oxidation of fat, the methods may be used to assess the degree of oxidation of these materials. Various methods for carrying out the thiobarbituric acid test are outlined and critically evaluated. Details are given of a simple spectrophotometric procedure for the determination of the thiobarbituric acid value. The fat (1 g ± 5 mg) is weighed into a test-tube and 10 ml of 0·01 *M* aq. 2-thiobarbituric acid soln., 5 ml of 0·7 *N* HCl and three small glass beads are added. The tube is closed with a condenser and the contents are heated gently and held at b.p. for 30 min. After rapid cooling of the contents to room temp., 5 ml of 20% trichloroacetic acid is added with shaking. The soln. is filtered and the extinction of the clear filtrate determined against a reagent blank. A calibration curve is constructed from extinction values obtained from a series of soln. of tetraethoxypropane in 40% ethanol after reaction with thiobarbituric acid as described above.

S. M. Marsh

4172. **Determination of fatty acid composition by gas chromatography and u.v. spectrophotometric methods.** L. Malin (Universal Oil Co., Ltd., Hull). *Perfum. Essent. Oil Rec.*, 1959, **50** (6) 505–507.—A review with 8 references.

4173. **Determination of the degree of rancidity of fats by a colorimetric method with *sym.*-diphenylcarbazide.** B. A. J. Sedláček and R. Rybin (Abt. f. Lebensmittelhygiene, Hygiene Inst., Prague). *Fette, Seif., Anstrichmitt.*, 1959, **61** (2), 134–138.—The reaction of rancid fats with *sym.*-diphenylcarbazide (**I**) suspended in CCl_4 to give a red colour, which can be examined spectrophotometrically or colorimetrically, is applied to the determination of the degree of rancidity. The fat (5 ml) and 5 ml of reagent (0·5 g of **I** suspended in 100 ml of CCl_4) are mixed in a calibrated test-tube (16 mm × 160 mm) and, after being heated for 4 min. in a water bath almost at boiling-point, is cooled and the level re-adjusted to 10 ml with CCl_4. It is then filtered in an apparatus protected from light and the extinction is measured at 550 mμ within 15 min. in a 16-mm cell. A blank sample of fat in CCl_4 is also examined to eliminate errors due to the colour of the fat. Lard or suet must be melted before being placed in the cell, and an additional 5 ml of CCl_4 added to the mixture after heating on the water bath. The degree of rancidity is determined by comparison of the extinction with a standard scale consisting of various soln. of Safranine O (C.I. Basic Red 2). Results are detailed for fats with various degrees of rancidity and compared with those obtained by other methods. Limiting values for the extinction are given by which butter, lard and oils may be classified as fresh, stored, slightly rancid, rancid or very rancid.

S. M. Marsh

4174. **Potentiometric determination of high molecular fatty acids and fatty acid mixtures.** C. Sass (REWO Chem. Fabrik G.m.b.H., Schlüchtern). *Fette, Seif., Anstrichmitt.*, 1959, **61** (5)

5.—Potentiometric titration of the potassium with $AgNO_3$ soln. is proposed for the identifica- and quant. analysis of fatty acids from *n*-octoic to stearic acid, and their mixtures. The acid le (0·12 to 0·25 g) is dissolved in 5 ml of n and titrated with 0·5 *N* ethanolic KOH st phenolphthalein and made up to 250 ml water. After warming the soln. to complete conversion into potassium salts, it is titrated 0·1 *N* $AgNO_3$ in 0·25-ml additions against a nel electrode. The temp. at which titration rried out depends on the acid. Typical titra- curves are shown. Results are accurate in 1% for single acids and within 3% for ures containing up to seven components. If than 25% of oleic acid is present, it cannot etermined. The presence of oleic and other turated acids increases the titration time use of deposition on the electrodes. Unknown ponents of an acid mixture may be identified irst titrating the mixture and then adding n amounts of various acids in turn and rving their effect on the titration curve.

S. M. Marsh

75. **Effect of varying the chemical composition ne stationary liquid on the resolution of the -chain saturated and unsaturated fatty acid s by gas - liquid chromatography.** S. R. Lipsky, . Landowne and M. R. Godet (Yale Univ. Med. New Haven, Conn., U.S.A.). *Biochim. hys. Acta*, 1959, **31** (2), 336–347.—A relation- is found between the relative solubility of rated and unsaturated acid esters in a particular nary phase and its chemical composition. separations attainable on different polyesters of ylene glycol (**I**) in relation to mol. wt. and ity are studied. On columns of Celite 545 d with a succinate polyester of **I** at 200°, ated and unsaturated fatty acid esters from o C_{26} are separated in 38 min. with He as r gas. Results obtained on a standard mixture thyl esters are discussed. W. H. C. Shaw

76. **Determination of fat-soluble vitamins in -dispersed preparations.** G. Cavina (Ist. Sup. à, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1957, 00–912.—By the described method fat-soluble ins can be recovered without saponification. *dure*—The aq. dispersion (2 ml) is mixed with Florisil (washed with 95% ethanol, dried at 100° and activated at 600° for 4 hr.) until powder is obtained. This is transferred to omatographic column (14 mm × 230 mm), luted with the selected solvent (peroxide-free l ether, benzene or *cyclo*hexane) at a rate of 0 per min. The determination is carried out e eluate after removal of the solvent, if ary, on a water bath in a current of N.

L. Zanoni

77. **Colorimetric method for the determination min A.** G. Cavina (Ist. Sup. Sanità, Roma, *R.C. Ist. Sup. Sanit.*, 1957, **20**, 913–922.— *t*—To a 20% soln. of $SbCl_3$ add of abs. ethanol and 2% of acetyl chloride. this soln. to 1% and add 2% of acetyl le. *Procedure*—To 1 ml of vitamin soln. in containing 0·5% of ethanol add 4 ml of t; set aside the soln. for 2 min. and measure tinction at 550 mμ against $CHCl_3$. The is stable for 10 min.; it is necessary to work to 25° in the dark. L. Zanoni

4178. **Study of the gravimetric tungstosilicate method of assay for thiamine.** E. E. Vannatta and L. E. Harris (Ohio State Univ., Coll. of Pharm., Columbus). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (1), 30–34.—The method of the B.P. 1953 (*cf.* Adamson and Handisyde, *Quart. J. Pharm. Pharmacol.*, 1948, **21**, 370) has been improved. Results of the application of the proposed procedure to thiamine nitrate or hydrochloride, alone or in tablets or soln., are in good agreement with those obtained by 3 other methods. Interference may be caused by high concn. of betaine, choline or nicotinamide, but not by other vitamins. *Procedure*—Heat the soln. of thiamine (25 to 50 mg) in dil. HCl (1:24) (50 ml) to boiling and rapidly add 10% tungstosilicic acid soln. (2·75 to 4 ml) drop by drop. Continue to boil gently with stirring for 3 min. Transfer the ppt. to a sintered glass crucible and wash with a mixture of dil. HCl - tungstosilicic acid soln. (50:1) (10 × 5 ml) and with dil. HCl (3 × 5 ml). Draw air through the crucible for 1 or 2 min. and wash the ppt. with acetone (2 × 5 ml). Dry the residue at 80° for 3 hr. and weigh.

A. R. Rogers

4179. **Gravimetric tungstosilicate method of assay for thiamine in the presence of interfering substances.** E. E. Vannatta and L. E. Harris (Ohio State Univ., Coll. of Pharm., Columbus). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (1), 34–36.—An ion-exchange method is used to separate thiamine (**I**) from products containing crude extracts and concentrates or formulating agents which interfere with the gravimetric assay. *Procedure*—Pass the soln. containing **I** (25 to 50 mg) at pH 4·5 at a rate of flow of 2 or 3 ml per min. through a column of Amberlite IRC-50 resin (10 g) buffered at a pH of 5·6. Wash the column with H_2O (15 ml) at a rate of 4 ml per min., and then successively at a rate of 5 or 6 ml per min. with H_2O (50 ml), acetone - H_2O (1:2) (15 ml), acetone - H_2O (2:1) (50 ml), acetone - H_2O (1:2) (15 ml) and H_2O (50 ml) and reject the washings. Elute **I** with 5% HCl until the eluate is acid to methyl orange and then with dil. HCl (1:24) (60 ml) at a rate of 1 ml per min. Assay the combined eluates by the tungstosilicate method (*cf. Anal. Abstr.*, 1959, **6**, 4178).

A. R. Rogers

4180. **Determination of thiamine and its phosphate esters.** A. Segre, C. Ciriello and M. Reviglio (Lab. Ric. Farmochim. Cutolo, Calosi, Napoli, Italy). *Gazz. Chim. Ital.*, 1957, **87**, 1199–1202.—An electrophoretic method is described for the determination of thiamine and its mono-, di- and tri-phosphates. *Procedure*—The sample (0·1 g) is dissolved in 10 ml of water and a 0·0015-ml aliquot is submitted to electrophoresis by Siliprandi's method (*Anal. Abstr.*, 1956, **3**, 260). The paper is dried and the spots are located with a Wood's lamp at 2600 A. The spots are cut out and placed in a test-tube with 5·5 ml of H_2SO_4 (0·1 *N*), 0·5 ml of acetate buffer (2 *M*), and 20 mg of taka-diastase. After incubation overnight at 45° the soln. is made up to a known vol.; 2 ml of this soln. is transferred to a 50-ml separating-funnel, with 3 ml of acid KCl soln. (to 1 litre of 25% aq. KCl soln. add 8·5 ml of conc. HCl) and 3 ml of oxidising soln. [to 1 ml of 1% aq. $K_3Fe(CN)_6$ soln. add 24 ml of 3·5 *N* NaOH] and 13 ml of butanol. This mixture is shaken for 2 min. The alcoholic phase is separated, dried with anhyd. Na_2SO_4 and the fluorescence is measured in a fluorimeter with Corning filter No. 5970, and

compared with that of a standard soln. containing 0·01 mg of thiamine hydrochloride per ml.
L. Zanoni

4181. **Assay of thiamine in biological fluids.** H. Baker, I. Pasher, O. Frank, S. H. Hutner, S. Aaronson and H. Sobotka (Mt. Sinai Hosp., New York, U.S.A.). *Clin. Chem.*, 1959, **5** (1), 13–17.—A microbiological method is described in which the protozoan *Ochromonas malhamensis* is used. This organism is suitably sensitive and has the advantage of requiring intact thiamine. It does not respond to, nor can it combine with, the thiazole or pyrimidine moieties.
H. F. W. Kirkpatrick

4182. **Chemical determination of riboflavine in foods. I. Analytical principle.** B. Gassmann and K. Täufel (Inst. f. Ernährung, Potsdam-Rehbrücke, Germany). *Nahrung*, 1958, **2**, 450–463.—Riboflavine is quantitatively converted to lumiflavine by treatment of riboflavine soln. with 0·75 to 3 *N* NaOH, followed by irradiation with a 250-W bulb for 15 min. The lumiflavine is extracted with $CHCl_3$ and is then determined photometrically. The lower limit of dilution is 0·0008 μg per ml.

II. Analytical methods. B. Gassmann and H. Plessing. *Z. LebensmittUntersuch.*, 1959, **109** (2), 135–145.—The method described for the extraction of riboflavine from milk products, plant material, yeast, etc., gives the same results as the more laborious alkaline hydrolysis and treatment with trypsin and phosphatase. The following operations must be carried out in subdued light. The comminuted sample (containing 15 to 100 μg of riboflavine) is autoclaved at 121° with 0·1 *N* HCl (150 ml) for 30 min. The mixture is then treated with diastase (≃ 2 g) at 45° and pH 4·5 to 5·4 for 30 min.; this treatment removes solids that occlude riboflavine. After filtration, the soln. is suitable for the fluorimetric determination of riboflavine as lumiflavine (*cf. Anal. Abstr.*, 1959, **6**, 2711, and Part I, above). Chem. Abstr.
P. S. Arup

4183. **Suitability of 2:4-dinitrophenylhydrazine methods for determining dehydro- and total ascorbic acid in raw and cooked vegetables. I. Dehydroascorbic acid.** J. Gordon and I. Noble (Univ., St. Paul, Minn., U.S.A.). *Food Research*, 1959, **24** (1), 1–5.—The 2:4-dinitrophenylhydrazine (**I**) deriv. prepared under the usual conditions (Roe *et al.*, *J. Biol. Chem.*, 1944, **152**, 511) for determining ascorbic acid (**II**) and dehydroascorbic acid (**III**) in vegetable extracts were purified by chromatography. Concn. of **II** determined from the fraction most closely resembling the **I** deriv. of **II** were lower than those found by the usual method. The spectrum of the blue fraction which served as a basis for calculating the concn. of **III** followed the general pattern of maxima and minima typical of the spectrum of **II**, but the max. at 530 mμ was smaller in proportion to the max. at 370 mμ. The concn. of **III**, based on the purified deriv. and corrected for interference due to reduced **II**, was lower than that found by the usual method for both cabbage and asparagus.

II. Total ascorbic acid. J. Gordon and I. Noble. *Ibid.*, 1959, **24** (1), 6–13.—Concn. of total **II** after reduction of **III** in cabbage and asparagus were determined with and without chromatographic separation. The results of the latter method were lower for asparagus, but not for cabbage; in cooked samples they were lower in both cases. N. E.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

4184. **Apparatus for the continuous determination of carbon dioxide in air.** L. Luria (Ist. Igie e Microbiol., Univ. di Torino, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1958, **34** (7), 353–354.—The air bubbled through 0·001 *N* $NaHCO_3$ containi phenol red in a vessel placed between a light sour at 590 mμ and a photo-electric cell connected to recording galvanometer. The increase in colour recorded on a chart and the CO_2 content is obtain by reference to a calibration curve. Each increme of 0·05% of CO_2 gives a reading with an error ± 0·002%. The delay in the recording is 7 to sec.
L. Zanoni

4185. **Determination of suspended matter water and its loss on ignition.** M. Enger. *Gas-Wasserfach*, 1958, **99**, 488–490.—Experiments a described on the use of glass-fibre paper, develop for chromatographic work, for the filtration water and the determination of suspended matte The suitability of the brittle fibre paper for use a filter, the possibility of obtaining const. wt. b drying and by ignition, and the filtration effe were tested. Asbestos Gooch crucibles were us for comparison. The glass-fibre filter was fou to give complete filtration and to maintain i efficiency with large amounts of water. For t determination of the amount of suspended matt in water, it was sufficient to wash and dry t filter; if residue and loss on ignition were also to b determined, the filter was ignited. The method simple and accurate and gives comparable result it is cheap and time-saving.
Water Pollut. Abstr.

4186. **Spectrophotometric determination dissolved oxygen in water.** B. D. La Mont a N. E. Gordon, jun. (Westinghouse Electric Co Atomic Power Dept., Pittsburgh). *U.S. Atom Energy Comm.*, Rep. WCAP–917, 1958, 13 pp. High-pressure boilers or nuclear power plan require an accurate determination of dissolved in feed and process water to minimise corrosio Improved sampling and analytical techniques the Winkler method are proposed. Hypoderm syringes are used to add the reagents to the sampl vessel to prevent atmospheric contamination. T liberated iodine is extracted with $CHCl_3$, and extinction of the soln. is measured and compa with a standard curve. Dissolved O in water c be determined in the concn. range of parts per or parts per 10^9. Nucl. Sci. Abstr.

4187. **Rapid potentiometric determination sulphate, nitrate and chloride ions in water and aqueous solutions.** A. Wagner. *Mitt. Grosskesselbesitzer*, 1958, (52), 50–53.—Depend on the salt content, from 20 to 200 ml of wate passed through a column containing about 60 of a cation-exchange resin (washed free from a after activation) to convert the salts into t corresponding acids. This soln. is then titra with 0·2 *N* $Ba(OH)_2$ to a pH of ≃ 6·85 (but > 7 the amount of $Ba(OH)_2$ corresponds to the sum SO_4^{2-}, Cl^- and NO_3^-. Add 40 ml of ethano *iso*propyl alcohol and titrate the soln. with 0· K palmitate to a pH of 7·65. The amount o palmitate soln. corresponds to the sum of the and NO_3^-. Replace the hydrogen electrode one of silver, add 5 ml of HNO_3 (*d* 1·20) to

., and titrate with 0·2 *N* $AgNO_3$ to a p.d. of mV (*vs.* the S.C.E.). The method of calculating concn. of the various ions is given. Phosphate (> 3 mg per litre) and CO_3^{2-} interfere with titration. Excessive amounts of PO_4^{3-} are oved by pptn. with basic lead carbonate at a ≃ 12·0 before passage through the cation resin. bonate ions are removed by passing CO_2-free through the soln. from the exchange resin. application of this method to the determination S in the products from washing bomb calori- ers is discussed. The method is very rapid gives results comparable with those from the al methods. CHEM. ABSTR.

188. **Determination of silicon in sea water.** B. Isaeva. *Trudy Inst. Okeanol. Acad. Nauk*, 8, **26**, 234–242; *Ref. Zhur., Khim.*, 1959, (4), str. No. 11,494.—A detailed study is presented the conditions for the photometric determination sea water of Si as the yellow molybdosilicic eropoly acid. It is shown that Na_2SiO_3 or SiF_6 soln., which are stable for 4 to 5 months, uld be used for standards instead of K_2CrO_4 or ric acid. The effect of salinity on the repro- ibility of the determinations is studied and it is wn that the soln. for calibration curves must be pared as for a sea water containing a minimum ount of SiO_2 and the same salinity as the water ng analysed. It is shown that the colour ensity of the molybdosilicic acid complex is pendent on the lapse of time between the addition ammonium molybdate and H_2SO_4 to the test n., owing to the rapid polymerisation of silicate s; hence the reproducibility of the results is versely affected. It is therefore recommended t a previously prepared mixture of ammonium lybdate and H_2SO_4 be added; 50 ml of test soln. uire 1·1 ml of a mixture (1:10) of 50% H_2SO_4 10% ammonium molybdate soln. The impor- ce of a strict check on the acidity of the test n. at the moment of complex formation is ssed. K. R. C.

189. **Determination of naphthenic acid in ural waters [containing humic acid].** E. A. s and A. N. Fikhman. *Novost. Neft. Tekhn. Zog.*, 1958, (7), 36–37; *Ref. Zhur., Khim.*, 1959, Abstr. No. 11,531.—The method is based on difference in behaviour of the two acids on tment with acidified H_2O, benzene or $CHCl_3$. mic acid is completely recoverable from H_2O as rown layer on top of the soln. and naphthenic dissolves in benzene and $CHCl_3$. *Procedure*— lify the sample (50 to 200 ml) with dil. H_2SO_4), agitate for 10 to 15 min. with $CHCl_3$ (2 × co 20 ml), filter the extracts through a glass under vacuum, wash the filter with $CHCl_3$ evaporate off the $CHCl_3$ from the combined ates. Dissolve the moist residue in 5% NaOH (1 to 3 ml), dilute the soln. and determine the nthenic acid concn. by a turbidimetric method. the filter with the humic acid for 30 to 60 min. 5°, and treat the residue with 5% NaOH soln. 3 ml), dilute the soln. and determine humic colorimetrically. Tests on synthetic mixtures ed that naphthenic acid can be recovered pletely. K. R. C.

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also Abstracts—**4047**, Determination of Ce^- in r. **4209**, Mol. distillation for organic particu- in air. **4210**, Recording of O content of waste s.

Agricultural analysis

Soil, fertilisers, weed-killers, pesticides, animal feeding-stuffs.

4190. **Use of the thermobalance in the analysis of soils and clays.** M. Schnitzer, J. R. Wright and I. Hoffman (Chem. Div., Canada Dept. of Agric., Ottawa). *Anal. Chem.*, 1959, **31** (3), 440–444.—The thermogravimetric behaviour of different soils, clays and inorganic carbonates has been examined, and possible errors in the determination of hygroscopic H_2O, organic matter and carbonates are discussed. Losses due to H_2O and readily volatile organic matter occur between 150° and 240°; values lie between those obtained by oven-drying at 105° and by the Karl Fischer titration. Organic matter is lost between 210° and 550°. Carbonate added to soil is quant. recovered, and it is possible to distinguish between magnesite and calcite or dolomite, but not between the last two compounds. In a mixture (1:1) of bentonite (H form) and illite both components can be identified; data for kaolinite are also given, and the determination of lattice H_2O in pure clays or simple mixtures seems possible. The presence of allophane, hydrated oxides of Fe or Al, or > 40% of clay causes errors in the determination of organic matter, and inaccurate values for carbonate are obtained in the presence of biotite or tremolite. Gypsum or hydrous silicates give high values for hygroscopic H_2O. P. D. PARR-RICHARD

4191. **Determination of boron in soil and plants. I. Colorimetric method.** F. Burriel-Martí, S. Jiminéz Gómez and M. Rodríguez de la Peña (Dept. Anal. Chem., Inst. Edafologia, Madrid). *An. Real Soc. Esp. Fis. Quim., B*, 1958, **54**, 35–42.—The carmine method for the determination of boron is studied and improved. The effects of temp., vol. of reagent, time of reaction and interference from other ions are considered and the following modified procedure is evolved. Take an aliquot (2 ml) of the sample soln., add conc. HCl (2 drops) followed by conc. H_2SO_4 (10 ml); shake well, cool and add carmine reagent [0·05% (w/v) in conc. H_2SO_4] (10 ml); shake well and read the absorption at 585 mμ. The Beer - Lambert law is obeyed and results are accurate to within ± 2%. Conditions for the production and storage of boron-free water are given. C. A. BLAU W. R. O'NEILL

4192. **Aluminium studies. Soil and plant analysis of aluminium by modification of the aluminon method.** T. L. Yuan and J. G. A. Fiskell (Dept. of Soils, Univ. of Florida, Gainesville, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (2), 115–117.—The determination of Al by reaction with aluminon in ammonium acetate buffer, to give a coloured complex, is specific and accurate if hydroxy-ammonium chloride is added to prevent interference by Fe. Details are given of the colorimetric properties of the complex, and the effects of pH, heating, and other ions on its extinction. The recovery of Al added to soil or plant digests was satisfactory. M. D. ANDERSON

4193. **Determination of carotene in fodder.** A. Daghetta and O. Bruss (Staz. Sper. del Freddo, Milano, Italy). *Ann. Sper. Agr.*, 1956, **10** (4), Suppl. CIX–CXIV.—A comparative study of methods of extracting and determining β-carotene is presented. The method advocated is that of Wall and Kelley (*Ind. Eng. Chem., Anal. Ed.*, 1943,

15, 18) with some modifications. *Procedure*—The dried, powdered fodder (4 g) is extracted in a Soxhlet apparatus with light petroleum - acetone (7:3). The soln. is evaporated at reduced pressure, and the residue is dissolved in light petroleum and made up to 50 ml; 20 ml is transferred to a chromatographic column (18 mm × 230 mm) packed with Al_2O_3 - Celite (1:1). The column is eluted with light petroleum and the extinction of the eluate is measured directly at 450 mμ. L. ZANONI

4194. **Analysis of substituted acetic acids used as herbicides. I. Spectrophotometric determination.** B. T. Milićević and S. Dj. Janković (Plant Protection Inst., Belgrade). *Bull. Soc. Chim., Belgrade*, 1957, **22** (5–7), 363–367.—Ultra-violet absorption curves of the soln. in diethyl ether of trichloroacetic acid (**I**), 2:4-dichlorophenoxyacetic acid (**II**), 2:4:5-trichlorophenoxyacetic acid (**III**) and 4-chloro-2-methylphenoxyacetic acid (**IV**), were all prepared in the 210 to 350-mμ range. It was established that for concn. of 1 to 10 mg per 100 ml of these acids there is a linear relation between concn. and the extinctions of the absorption maxima and that this relation can be utilised for their quant. determination. In the presence of **I** any of the three chlorophenoxyacetic acids can be quant. determined.

II. Polarographic determination. B. T. Milićević and M. M. Mišić. *Ibid.*, 1957, **22** (5–7), 369–372.—Polarographic determinations of **I**, **II**, **III** and **IV** were investigated and it was established that **I** can be determined in the presence of **II** within the experimental error for the estimation of pure **I**. A newly developed method, with tetramethylammonium iodide as an auxiliary electrolyte, was used for the polarographic determinations of **II**, **III** and **IV**.

III. Refractive index method. B. T. Milićević and O. D. Vitorović. *Ibid.*, 1957, **22** (5–7), 373–375.—The concn. of **I**, **II**, **III** and **IV** in soln. in diethyl ether were determined by the use of a refractometer. A method for the determination of mixtures of **I** and **II** in ether soln. was developed. After the refractive index reading, the ether is evaporated and the acid ratios are calculated from the residue by means of the standard triangle.
A. GROCHOWSKI

4195. **Detection and estimation of parathion residues in foodstuffs.** S. N. Mitra, P. N. Sen Gupta and B. R. Roy (Cent. Food Lab., Calcutta). *J. Instn Chem., India*, 1958, **30** (6), 321–325.—The method of Gunther and Blinn ("Analysis of Insecticides & Acaricides," Interscience Pub., Inc., N.Y., 1955, Vol. VI, p. 445) for determining parathion (**I**) residues is found to give 99% recovery of added **I**, and to be simpler than that recommended by the A.O.A.C. Both methods are based on extraction of the insecticide with benzene, reducing the NO_2 group to NH_2, diazotising, and coupling the resulting water-sol. compound with N-1-naphthylethylenediamine to develop a magenta colour. Since the colour reaction may be given by other extractable organic compounds as well as **I**, the presence of **I** in the benzene extracts was confirmed by identification of P and S. Benzene may extract organic P from certain foodstuffs (*e.g.*, wheat atta), but rarely extracts organic S.
M. D. ANDERSON

4196. **Determination of dimeton-methyl residues in plant material.** E. Q. Laws and D. J. Webley (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1959, **84**, 28–32.— representative sample (50 g) of the material macerated with 100 ml of aq. methanol (1 + then filtered, and the residue is washed and t combined filtrate and washings are extracted w light petroleum, the extracts being discarded. T aq. layer is extracted with $CHCl_3$ and the extra are evaporated to a small vol. When this procedu gives a high blank value the initial maceration made with *iso*butyl alcohol, and the filtrate is mix with light petroleum and extracted with wat The combined extracts are washed with lig petroleum and then extracted with $CHCl_3$ as befo The $CHCl_3$ extract is applied to a chromatograph column of active carbon in $CHCl_3$ and the colur is eluted with $CHCl_3$. The eluate is evaporated dryness and the residue is dissolved in water. maceration with *iso*butyl alcohol was used, t eluate must, for safety, be treated with cor HNO_3 and conc. HCl and boiled to dryness befc adding HNO_3, HCl and $HClO_4$, as in the meth of Field and Laws (*Analyst*, 1957, **82**, 667). T phosphorus content is then determined by spectr photometric measurement of the molybdenum bl complex. By the method described, two isome (thionate and thiolate) and two oxidation produc (a sulphoxide and a sulphone) are extracted $CHCl_3$, but a sulphonium compound is not.
A. O. JONES

4197. **Infra-red procedure for quantitative dete mination of residues of 2:4:4′:5-tetrachlor diphenyl sulphone (Tedion) on and in citrus-fru peel.** F. A. Gunther, R. C. Blinn and J. Barkley (Univ. of California Citrus Exp. Sta Riverside, U.S.A.). *J. Agric. Food Chem.*, 195 **7** (2), 104–105.—Methods for total Cl^- can be us to establish the maximum residues of the acarici Tedion (**I**) remaining on treated citrus fruit but, order to investigate the behaviour of degradati products, the more specific i.r. method w developed. Chromium trioxide in glacial acetic ac to which Tedion is stable, is used to destroy int fering substances extracted with **I** by $CHCl_3$ fr citrus peel. The purified **I** dissolved in CS_2 is th determined by measurement of the strong sulphc absorption peak at 1160^{-1} cm; 20 μg of **I** can determined in 100 g of peel. M. D. ANDERSON

4198. **Determination of phenkaptone residues fruit and vegetables.** H. J. Hardon, H. Brun and E. W. van der Pol (Keuringsdienst van War Keizersgracht 732–734, Amsterdam, Netherlan *Analyst*, 1959, **84**, 102–105.—In the proced described for the determination of residues of acaricide S-(2:5-dichlorophenylthiomethyl) OO ethyl phosphorothiolothionate (phenkaptone) fruit and vegetables, the sample is extracted w light petroleum, the solvent is removed and residue is dissolved in a known vol. of met cyanide and the soln. is filtered. Plant waxes thus removed. An aliquot of the filtrate is hea under reflux with 8 *N* NaOH until all the me cyanide is hydrolysed and is then steam-disti with H_2SO_4, the distillate being collected in dichl methane. The organic layer, together with dichloromethane extractions of the aq. layer filtered through Na_2SO_4, and evaporated mercuric acetate in glacial acetic acid which is ad to prevent volatilisation of 2:5-dichlorothioph This is brominated with a soln. of Br in acetic and the excess of Br is removed with phenol, H is added and then Aldridge's reagent (a mixtur 10 ml of conc. HCl, 30 ml of pyridine and 70 r

ter added to a 5% soln. of benzidine hydro-
oride in 2% HCl). The extinction of the red
npounds formed by reaction of CNBr with
lridge's reagent is measured at 535 mμ and
erred to a calibration graph. The sensitivity of
method is 0·05 p.p.m. A. O. Jones

4199. **Chemistry of polyhalocyclopentadienes and ated compounds. XIV. The quantitative deter-nation of [the insecticide] Thiodan [6:7:8:9:-:10-hexachloro-1:5:5a:6:9:9a-hexahydro-6:9-thano-2:4:3-benzodioxathiepin-3-oxide].** R. emschneider and J.-C. Hilscher (Freien Univ., rlin, Germany). *Z. anal. Chem.*, 1959, **165** (4), 8–280.—The method is based on the reduction of Thiodan sulphur in acid medium to H_2S and separation as CdS. The concn. range is 17 to mg and the mean error for a single determina-n is ± 1·14%. The reducing agent used is zinc wder. For larger quantities of Thiodan (60 to 0 mg), oxidation with H_2O_2 in alkaline medium recommended; the SO_4^{2-} produced are pptd. th benzidine sulphate and are titrated. The ror is ≃ ± 2%. G. P. Cook

4200. **Determination of Phygon [2:3-dichloro-4-naphthaquinone] residues on food crops.** J. R. ne (Naugatuck Chemical, Div. U.S. Rubber Co., augatuck, Conn.). *J. Agric. Food Chem.*, 1958, 10), 746–747.—The colorimetric method described the determination of microgram residues of the ngicide Phygon (**I**) in food crops is based on the ecific reaction of **I** with dimethylamine to pro-ce an intense orange colour, which is measured 495 mμ. The method is sensitive to 1 μg per , and 0·5 μg per ml is detectable. The recovery **I** added to 5 food crops in amounts of from 0·4 1·0 p.p.m. averaged 91%. Interference values untreated samples averaged 0·02 p.p.m. of **I**. M. D. Anderson

4201. **Determination of warfarin in rodenticides.** Armstrong (Thos. Harley Ltd., Rodine Works, th, Scotland). *Chem. & Ind.*, 1959, (5), 154–5.—For warfarin concentrate 0·5%, the standard A.F.F. procedure can be used. Add 0·25 g of nple to 100 ml of *iso*propyl alcohol containing (v/v) of acetic acid. Shake the mixture for min. and centrifuge until clear; measure the inction at 271, 282 and 305·5 mμ, a similar ract of the oat-flour alone being used as a blank. e extinction (E) at 305·5 mμ × 1·13 = warfarin). The two other values of E at 271 and 282 mμ be used as a qual. check. For concentrates ed on maize-starch or china-clay, no blank is uired. Ethanol can be used instead of *iso*-pyl alcohol for samples containing warfarin in ta. W. J. Baker

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See also Abstract—**3898**, Hg in seed-treating erials.

5.—GENERAL TECHNIQUE AND APPARATUS

General

4202. **Microchemical apparatus. Part II:1953. Combustion boats, sheath and contact stars for microchemical analysis.** British Standards Institution (2 Park St., London, W.1). B.S. 1428: Pt II:1953. Amendment No. 1, 15.5.1959.—The title is changed to 'Combustion boats and sheath for microchemical analysis' and changes in the design of boats and sheaths are made.

4203. **Density hydrometers for use in milk.** British Standards Institution (2 Park St., London, W.1). B.S. 734:1955. Amendment No. 2, 17.4.1959.—Table 4 in Appendix D is withdrawn.

4204. **Burettes and bulb burettes.** British Standards Institution (2 Park St., London, W.1). B.S. 846:1952. Amendment No. 3, 16.4.1959.—Additions to the recommended dimensions are made.

4205. **Burettes with pressure-filling device and automatic zero.** British Standards Institution (2 Park St., London, W.1). B.S. 1428: Part D1: 1952. Amendment No. 1, 20.3.1959.—Additions to the recommended dimensions are made.

4206. **Circulation apparatus for photometric titrations.** C. Rehm, J. I. Bodin, K. A. Connors and T. Higuchi (Sch. of Pharm., Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 483.—A simple, inexpensive titration unit, which is particularly useful if a closed system is required, is described. The unit can be fitted with little modification to almost any design of spectrophotometer. K. A. Proctor

4207. **Improvements in the Kjeldahl method.** N. F. Komshilov. *Izv. Karel'sk. i Kol'sk. Fil. Acad. Nauk*, 1958, (3), 137–138; *Ref. Zhur., Khim.*, 1959, (5), Abstr. No. 15,112.—A special condenser, in the form of a tube with a globular extension, was fitted into the neck of the Kjeldahl flask which was maintained at an angle of 30° in the heater. The condenser prevented the evaporation of H_2SO_4 and was itself charged with conc. H_2SO_4. With this arrangement, ≃ 30 mg of test substance and 3 to 4 ml of H_2SO_4 were required. K. R. C.

4208. **Vapour take-off still-head. Improvements in laboratory fractionation of small quantities at reduced pressures in packed columns.** R. A. Hall (I.C.I. Ltd., Bothwell St., Glasgow, Scotland). *Anal. Chem.*, 1959, **31** (3), 437–439.—The still-head described is of all-glass construction, has no metal or moving parts and requires no grease. It is very suitable for small-scale work, particularly at reduced pressures with reactive materials. Good fractionating efficiency can be achieved at low pressures with columns constructed in the form of a narrow annulus, packed with helices constrained to the vertical plane. The purification and use of hexadec-1-ene in test mixtures are discussed. K. A. Proctor

4209. **Molecular distillation unit for organic particulates in air.** G. R. Sanders and H. L. Helwig (Div. of Lab., California State Dept. of Public Health, Berkeley, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 484–485.—The capacity of the unit described is such that 1 mg to 5 mg of material may be handled. Fraction collecting is simple and precise, and temp. and pressure are measured in an absolute manner. The distillation chamber, which is easily dismantled and cleaned, can be exhausted from the bottom, thus preventing any of the distillate from being entrained in the vacuum system. K. A. Proctor

4210. **Apparatus for recording the oxygen content of waste waters.** E. Ebbinghaus. *Wasserwirtschaft, Stuttgart*, 1958, **48**, 320–321.—Difficulties arise in the use of electrochemical methods for measuring the O content of river waters and waste waters because of uncontrollable variations in pollution. Suitable apparatus exists for measurement and recording the O content of a gas and the author describes the use of this method for water. In principle the method depends on exchange between equal vol. of water and O-free N. These are brought into contact in counter-current in a de-gasifying column and the O content of the withdrawn gas is determined and recorded. The accuracy of the method and its use in practice are described. WATER POLLUT. ABSTR.

See also Abstracts—**4946**, Moisture measuring apparatus.

Chromatography, ion exchange, electrophoresis

4211. **Partition chromatography and its use in the plant sciences.** J. F. Thompson, S. I. Honda, G. E. Hunt, R. M. Krupka, C. J. Morris, L. E. Powell, jun., O. O. Silberstein, G. H. N. Towers and R. M. Zacharius (U.S. Plant, Soil and Nutrition Lab., Ithaca, N.Y.). *Botan. Rev.*, 1959, **25**, 1–263.—A comprehensive review of chromatographic techniques, with applications for org. acids, sugars, phosphorylated compounds, amino acids, proteins, alkaloids, purines, auxins and pigments, is presented. (1166 references.) CHEM. ABSTR.

4212. **Separation of the elements of the second and third analytical groups by two-dimensional paper chromatography.** M. B. Čelap (Kemiski Inst., Belgrade, Yugoslavia). *Bull. Soc. Chim., Belgrade*, 1957, **22** (5–7), 345–352.—A procedure has been developed for the separation of Sb, Cu, Bi, As, Cd, Sn and Hg (group-2 elements) from Al, Ni, Cr, Mn, Co, U, Fe and Zn (group 3) by the use of two-dimensional ascending paper chromatography, avoiding preliminary separation of these elements into smaller groups. *iso*Butyl alcohol - HCl - water (82:3:15) was used as the first solvent, and pyridine - water (3:2) as the second solvent for the group-2 elements, the separations taking 16 and 4 hr., respectively. The spots were detected with H_2S and 2 *N* $AgNO_3$. For the group-3 elements, the first and the second solvents and the separation times were acetone - 6 *N* HCl (9:1) (4·5 hr.), and acetic acid - methanol (3:1) (10 hr.). The paper was sprayed with a mixture of salicylaldoxime and dimethylglyoxime in ethanol and then with soln. of dithizone in $CHCl_3$ and alizarin in ethanol for detection of the spots.

A. GROCHOWSKI

4213. **Multiple pH levels in chromatograms.** A. A. Bitancourt and A. P. Nogueira (Plant Cancer Res. Centre, Inst. Biol., São Paulo, Brazil). *Science*, 1959, **129**, 99.—Longitudinal lines are drawn on the paper and buffer soln. of different pH values are applied from pipettes along each line. The sample soln. is applied transversely and chromatographed. The effect of different pH values on the R_F values, colour, fluorescence and other properties of the substances present can then be studied. The application to decomposition products of indol-3-ylacetic acid is described.

H. F. W. KIRKPATRICK

4214. **Two-dimensional paper-chromatographic method combining ion-exchange and partition techniques.** C. S. Knight (W. and R. Balston Ltd., Springfield Mill, Maidstone, Kent, England). *Nature*, 1959, **183**, 165–167.—The development of a range of Whatman ion-exchange celluloses allows two techniques to be combined on one material in paper form. A description is given of applications of phosphorylated, carboxylated and aminated celluloses to the separation of metal ions.

H. F. W. KIRKPATRICK

4215. **Device for isolation of components separated by gas chromatography.** J. M. Lesser (Barrett Div., Allied Chemical Corp., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (3), 484.—The device described, in which the component is condensed in a collector-tube by means of solid CO_2, is very simple and can be used with any gas-chromatographic apparatus in which the effluent-gas exit is close to the detector or in which the gas-exit line is heated to prevent condensation.

K. A. PROCTOR

4216. **Applications of infra-red spectroscopy: the identification and determination of gas-chromatographic fractions.** D. M. W. Anderson (Dept. of Chem., The Univ., Edinburgh). *Analyst*, 1959, **84**, 50–55.—A simple apparatus for the collection of products separated from mixtures by gas chromatography and the application of vapour phase i.r. spectroscopy to their determination are described. The method is applicable to all vapours and liquids of low or medium b.p. The sample is collected by condensation in a specially designed trap immersed in liquid N, and is transferred to an evacuated gas cell at 0·5 to 1 torr, the pressure being subsequently brought to atmospheric by admission of an i.r. inactive gas, the partial pressure of the sample vapour being then $\simeq 1$ torr. For unheated cells the limit of b.p. is $\not> 175°$ to 180°. Identification and determination are made by comparison with spectra for standard vapours, and a manometric apparatus is described for the accurate dilution of vapours in the gas cell. Amounts of $\simeq$ 5 μmoles can be identified and quant. determined to within $\pm$ 2%.

A. O. JONES

4217. **Thermistor detectors in gas chromatography.** W. K. R. Musgrave (Chem. Dept., Univ. of Durham, England). *Chem. & Ind.*, 1959, (2) 46.—Two thermistors (Type A or I) are sealed into a brass block by means of a cold-curing silicone rubber. Pairs of thermistors of 1000, 2000 and 500,000 ohms have been used up to 220°. In comparison with hot-wire thermal-conductivity detectors, these detectors have a far lower noise level, particularly at higher temp.

K. A. PROCTOR

4218. **Zone electrophoresis. Some basic considerations in design.** N. H. Martin (Dept. of Chem. Pathol., St. George's Hospital, London). *Analyst*, 1959, **84**, 89–94.—The basic requirements for the efficient application of zone electrophoresis as distinct from free-boundary electrophoresis, are discussed, with details of the provisions that ensure stable electrical conditions across the field during analysis, stability of the buffer medium and its maintenance at an even concn. during analysis. An electrophoretic cell with a filter-paper strip as the stabilising medium is described and illustrated.

A. O. JONES

4219. **Combined paper- and agar-electrophoresis apparatus.** W. F. Bon and J. de Vries. *Rec. Trav. Chim. Pays-Bas*, 1959, **78** (2), 173–176 (in English).—The apparatus (illustrated), constructed of Perspex, permits simultaneous electrophoresis on paper and agar. Two separate electrode and buffer systems are arranged at right angles to each other. Conditions for the separation of serum proteins are given. D. G. Moss

Optical

4220. **X-ray analysis technique for very high temperatures.** E. Aruja, J. H. Welch and W. Gutt (Building Res. Sta., Watford, Herts., England). *J. Sci. Instrum.*, 1959, **36** (1), 16–20.—Constructional details are given of a powder-diffraction instrument in which the sample is mounted directly on the tip of a thermocouple, which also serves as heater. A maximum working temp. of about 1850° is possible with 20% Rh - Pt/40% Rh - Pt couples. G. Skirrow

4221. **Modification to the Unicam SP500 spectrophotometer for single-beam recording.** D. D. Shrewsbury (The Midland Tar Distillers Ltd., Res. Dept., Four Ashes, nr. Wolverhampton, England). *Analyst*, 1959, **84**, 68–70.—By means of the apparatus described, the wavelength control and the slit-width control of the Unicam SP500 spectrophotometer are rotated at such rates that equal wavelength intervals are scanned in as nearly equal time intervals as possible. The output from the amplifier of the instrument operates a potentiometer pen recorder. The mechanical and electrical details of the modification are described and two graphs are given as examples of its performance. With the attachments described, the instrument can be used either as a recording instrument or in the conventional manner. A. O. Jones

4222. **Use of the "singing" arc for increasing the reproducibility of quantitative determinations by the spectrographic method.** Yu. I. Belyaev and É. E. Vaĭnshteĭn (V. I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (1), 133–134.—The reproducibility of spectrographic determinations is improved by using a d.c. "singing" arc, *i.e.*, by including the arc in an oscillating circuit to stabilise the discharge; if the frequency is 50 to 1,000 c/s the arc begins to emit sound. The stabilising effect is demonstrated by a comparison of the distribution of errors in determining Sb and Pb in uranium oxide by the singing d.c. arc and the normal d.c. arc. C. D. Kopkin

4223. **Background correction and evaluation by means of a projectable "graphicon" for spectral analysis.** T. Török, M. Kovács and S. Erdélyi (Inst. for Inorg. and Anal. Chem., L. Eötvös Univ., Hungary). *Z. anal. Chem.*, 1959, **165** (4), 241–247.—The procedure, which is essentially a further development of the graphical method of Hurwitz (*Anal. Chem.*, 1953, **25**, 1028), is described and a scheme for background correction and evaluation given. Practical examples are shown. B. B. Bauminger

4224. **Simple recording double-beam photometer for use with conventional spectrophotometers.** G. Allandi and G. S. Crescenzi (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1957, **20**, 105–112.—A simple and inexpensive recording double-beam photometer which can be attached to different types of non-recording spectrophotometer is described. Dimensions and constructional details are given. This apparatus can be used for the analysis of chromatographic eluates. L. Zanoni

4225. **Sapphire-windowed spectrophotometer absorption cell for use with corrosive liquids.** E. Raisen (Armour Res. Found., Illinois Inst. of Technol., Chicago, U.S.A.). *Anal. Chem.*, 1959, **31** (3), 485–486.—The cell described can be used with the Beckman DU spectrophotometer or any other instrument with a similar cell carrier. The cell can be used in the presence of fuming HNO_3 and HF, and the sapphire windows have optical and physical properties slightly superior to those of quartz. K. A. Proctor

4226. **Infra-red high-resolution grating spectrometer.** E. K. Plyler and L. R. Blaine (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand.*, 1959, **62** (1), 7–9.—The instrument described can be used as either a single- or double-pass spectrometer and gives partial resolution of 0·025 cm^{-1} in the region from 1600 to 3500 cm^{-1}. The spectra can be scanned at speeds from 2·5 to 200 min. of time per degree of rotation. Lines separated by 0·05 cm^{-1} are completely resolved and the fine structure of some bands in the mol. spectra of CH_4, C_2H_4, NH_3 and similar compounds can be elucidated. W. J. Baker

4227. **Oscillating-plate differentiator for spectrophotometry.** I. G. McWilliam (I.C.I., Ltd., Blackley, Manchester, England). *J. Sci. Instrum.*, 1959, **36** (1), 51–52.—A Grubb-Parsons S4 i.r. monochromator has been modified to give derivative spectra over the wavelength range 2 to 15 μ by placing a 1·0-mm sodium chloride plate in the light path and oscillating it at 750 oscillations per min. through an angle of 3°. This results in a modulation amplitude of approx. 0·005 μ at 3 μ. G. Skirrow

4228. **Mechanical auxiliary recorder for an infra-red spectrometer.** P. R. Watt (Vitamins Ltd., Dorking Rd., Tadworth, Surrey, England). *Chem. & Ind.*, 1959, (2), 44–45.—The simple and inexpensive recorder described is designed for use with the Grubb-Parsons DBS4 double-beam spectrometer and has given trouble-free service for over 6 months. It is used to provide small copies of i.r. spectra for punched-card indexing. The apparatus could be adapted for use with other spectrometers in which the servo motor or pen-drive mechanism is reasonably accessible. K. A. Proctor

4229. **Convenient infra-red standards in the carbonyl region.** G. T. Newbold (Roy. Coll. Sci. Tech., Glasgow). *Chem. & Ind.*, 1959, (5), 155.—A method of preparing thin films of poly(methyl methacrylate) (**I**) and poly(vinyl acetate) (**II**) supported on rock-salt plates is described. Both these supported films show symmetrical CO bands, **I** at 5·781 μ (1730 cm^{-1}) and **II** at 5·752 μ (1738 cm^{-1}). These values, which are corrected to vacuum and are accurate to $\pm$ 0·005 μ, are recommended as standards when calibrating double-beam i.r. spectrometers. They provide a standard band nearer to those shown by esters and ketones than are the bands of polystyrene now in use. W. J. Baker

4230. **Raman spectroscopy. II. Experimental technique for the measurement of vibration Raman spectra.** B. Schneider and K. Ulbert (Chem. Inst., Acad. Sci., Prague). *Chemie, Prague,* 1958, **10** (12), 961–974.—The construction of apparatus for the measurement of Raman spectra in laboratories possessing a suitable spectrograph and excitation source is described. The principles and a detailed description of the technique are given. J. ZÝKA

4231. **Simplified recording photometer for flame analysis.** R. H. Dreisbach (Stanford Univ. Sch. of Med., Calif., U.S.A.). *Anal. Chem.,* 1959, **31** (3), 479–480.—In the equipment described, the output of a photomultiplier (RCA IP21 from 200 to 700 mμ, Dumont 6911 up to 1200 mμ) is connected directly to the input of a chopper-amplifier potentiometric recorder (Varian Model G-10, 100 mV, 0·5 megohm input). Drift and random variations at max. sensitivity did not exceed ± 3% over a period of 8 hr. Detection limits are about 0·0001 p.p.m. of Na, 0·008 p.p.m. of Ca and 0·0034 p.p.m. of K. The accuracy and precision are comparable with those of similar equipment.
K. A. PROCTOR

4232. **Automatic recording in serial colorimetric analysis.** G. L. Miller and C. L. Cummings (Quartermaster Res. and Engng Center, U.S. Army, Natick, Mass., U.S.A.). *Anal. Chem.,* 1959, **31** (3), 481–483.—The device described provides point recording at a fixed wavelength, uniform spacing of points along the recorder chart and push-button control for intermittent operation. The reproducibility of the distance of pen travel is ± 0·003 at extinctions of 0·1 and 0·9 and ± 0·013 at an extinction of 1·7. K. A. PROCTOR

Thermal

4233. **Micro-method for the determination of boiling-point with direct temperature measurement.** H. Böhme and R.-H. Böhm (Pharm. Chem. Inst., Univ. Marburg/Lahn, Germany). *Mikrochim. Acta,* 1959, (2), 270–273 (in German).—An apparatus is described by means of which it is possible to determine the b.p. of 0·05 ml of substance with a mean error of ± 0·2°. Twenty-five compounds (with b.p. ranging from 34·5° to 238·3°) have been successfully measured. B. B. BAUMINGER

4234. **Refinements of the ring-oven method.** H. Ballczo (II Chem. Inst., Univ. Vienna, Austria). *Mikrochim. Acta,* 1959, (2), 314–317 (in German).—The metal ring-oven has now been replaced by one of glass, thus eliminating suspected interference in the analysis of metals. The oven is heated by the vapours of organic liquids which are boiled in a 100-ml flask and the vapours are passed through the oven to a condenser. An oven temp. of 105° to 110° is attained with *sym.*-tetrachloroethane. The improvements are discussed and details of the oven and accessories given. B. B. BAUMINGER

4235. **Apparatus for micro-thermogravimetric analysis with automatic recording of results.** S. S. Shchedrovitskiĭ, B. M. Moiseev and E. V. Mashintsev (N. S. Kurnakov Inst. of Gen. and Inorg. Chem., Acad. Sci., USSR). *Zavod. Lab.,* 1959, **25** (1), 122–125.—The apparatus for micro-work is described and illustrated. G. S. SMITH

Electrical

4236. **Rapid electrogravimetric separations by the use of Winkler electrodes. I.** M. S. Jovanović and S. Ž. Kostić (Kem.-Tehn. Zavod, Belgrade, Yugoslavia). *Bull. Soc. Chim., Belgrade,* 1957, **22** (5–7), 353–362.—Modified Winkler electrodes were successfully applied to separations of Ag from Cu, of Cu from Zn, Cd and Sb, of Sb from Sn, and of Bi from Sn, and the analytical procedure was developed. While Fischer-type electrodes are suitable for determinations of Sb in hydrolysed soln. of $Sb_2(SO_4)_3$, separations in which the modified Winkler electrode is used require excess of H_2SO_4 in the soln. to avoid hydrolysis.
A. GROCHOWSKI

4237. **Reproducibility of polarographic measurements.** F. von Sturm (Forschungslab. Siemens-Schukertwerke A.-G., Erlangen, Germany). *Z. anal. Chem.,* 1959, **166** (2), 100–114.—With detailed attention to conditions, reproducibility of half-wave potentials is ± 0·2 mV; for micro determinations utilising a three-electrode technique it is ± 1 mV; and for maximum potentials in square-wave polarograms ± 2 to 10 mV. The effects of depolarising substances and of the properties of the dropping electrodes and particularly of the capillaries are discussed.
J. P. STERN

See also Abstracts—**3877**, Isotopic analysis of lithium by mass spectrometry. **4056**, ^{14}C analysis for clinical studies.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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